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CHEMICAL LECTURE EXPERIMENTS

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CHEMICAL LECTURE EXPERIMENTS

BY

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INSTRUCTOR IN CHEMISTRY IN WESLEYAN UNIVERSITY

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To the Memory of

JOSIAH PARSONS COOKE

FOR FORTY-THREE YEARS ERVING PROFESSOR OF CHEMISTRY
AND MINERALOGY IN HARVARD UNIVERSITY

THIS BOOK IS DEDICATED

AS A TRIBUTE TO AN INSPIRING TEACHER

THE AUTHOR

PREFACE

The demonstration of chemical phenomena on the lecture table, though originally strongly imbued with an element of mysticism inherited from the hermetic art, has always been considered an essential phase of chemical instruction. After chemistry was placed on a broader basis by Lavoisier, and the black art converted to a science, experimental lectures still remained a legitimate factor in presenting the subject. Soon text-books and class recitations and later laboratory exercises for the student supplemented the lecture, and with the introduction of the latter a tendency to neglect the experimental lecture developed. Laboratory exercises, however great their influence in developing the experimental side of teaching the science, have their limitations, experimentally and educationally, and cannot supplant the experimental lecture, for it is in the lecture, and there only, where each experiment stands out clearly defined and unattended by the distractions necessarily accompanying laboratory exercises, that the first accurate observations of chemical phenomena can be made by students.

The late Professor Josiah P. Cooke, though one of the first to introduce laboratory exercises into the educational institutions of this country, said, nevertheless, "Experimental lectures are, I am convinced, much the best way of presenting these subjects [Chemistry and Physics] as systematic portions of knowledge." His belief in the method gave rise to that grand series of experimental lectures

delivered for more than forty years at Harvard College; a course of lectures whose stimulus to chemical study and research has influenced so many of the prominent teachers of chemistry in this country.

The most versatile and fluent chemical lecturer of his day, the late Professor Victor Meyer, was another great advocate of the experimental lecture. The breadth and scope of his lectures gave unusual opportunities for experiments, and his contributions to experimental science were of so brilliant and striking a character as to infuse new life into the chemistry of the lecture room.

Constant attendance on Professor Cooke's lectures for five successive years gave the first impulse to the preparation of this book, an impulse which was, through my intercourse with Professor Meyer, stimulated into a purpose.

The object of this book is primarily to furnish teachers with a large number of reliable lecture experiments. That these experiments require in many cases different treatment from those performed in the laboratory will be obvious when it is considered that the demonstrations on a lecture table must be of sufficient magnitude and of a character marked enough to enable the phenomena to be observed at as great a distance as possible.

The two or three previous works treating of this branch of experimental science are remarkably complete and exhaustive, but there is one serious hindrance to their ready adoption in most colleges and schools, *i.e.*, the requirement of elaborate and costly apparatus and the assumption of great proficiency on the part of the teacher in glass-blowing and general manipulation. To overcome this difficulty all experiments requiring especially elaborate apparatus have here been rigorously excluded. By adhering to this rule some familiar experiments have doubtless been omitted, but it has been possible in many cases to substitute an

equivalent experiment. As most of the apparatus required is that commonly used by students, it is considered that the large majority of experiments here presented may be easily performed with an ordinary laboratory equipment.

As an aid to the inexperienced the descriptions of the apparatus and the manipulations are given in considerable detail together with many suggestions regarding the preparation and care of apparatus. Experimental manipulation is not, however, merely a matter of rules, and, while the suggestions given will, it is hoped, prove of value to the inexpert, the fullest confidence so essential to successful experimenting must be obtained by experience.

With but few exceptions the elements are treated in the order of their arrangement in the periodic classification. An attempt has been made to have the cross-references in the index so complete that a list of experiments on any subject may be readily obtained.

The material here presented has been prepared with reference also to its use by students desiring collateral reading in connection with experimental lectures. As an elaboration of the laboratory manual, the book may also be used by students for the preparation of many compounds not considered in elementary text-books.

The great number of contributors to the evolution of the phase of experimental chemistry treated in this book renders it impossible to designate with any degree of justice the originators of the very large majority of experiments of this class, and rather than encumber the book with multitudinous references I prefer personally to make no especial claim to originality. My indebtedness to the former works pertaining to this subject, all of which have been freely drawn upon, is inadequately expressed by enumeration in the list on p. 419.

In performing the experiments, which have all had my

personal supervision, I have enjoyed the skilful assistance of Drs. H. M. Smith and J. F. Snell and Mr. W. S. Baker. I am deeply indebted to Professor W. P. Bradley, who has rendered me signal service in the many suggestions born of his long experience in chemical manipulation. I would also express my thanks to Professor W. O. Atwater, who generously placed his magnificent chemical library at my disposal. The index was prepared by Mrs. Cornelia Golay Benedict, whose assistance in this and in many other ways has made the writing of this book possible.

The object of this book will have been attained if it supplements to some extent the admirable works of Arendt, Heumann, and Newth in their task of stimulating the demonstration of chemical phenomena.

F. G. B.

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Middletown, Connecticut.

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CHEMICAL LECTURE EXPERIMENTS

INTRODUCTION

Owing to the independent nature of each experiment the description is in most instances prefaced with a short statement of the nature of the reaction under consideration. The equations have been given as far as practicable, and where the reaction is not regular and the product is of a complex nature, the fact that the equation represents the general reaction only is indicated by its being followed by an interrogation mark enclosed in brackets.

To facilitate the preparation of experiments, a list of the apparatus and the chemicals required has been appended to experiments demanding anything not ordinarily found on a lecture table. Just how extensive such a list should be has been a matter of considerable question, and to avoid undue repetition it is assumed that burners, test-tubes, retort stands, pneumatic trough, and such general apparatus, as well as the common reagents, are either on the lecture table or within reach.

Where approximate quantities of solid substances are required, it has been the custom hitherto to make reference to the size of a "pea," "walnut," "hazelnut," "egg," and so forth. The latitude thereby allowed in the selection of the quantity of material is often much greater than that intended by the writer, owing to the variations in the conceptions of the different sizes by individual experimenters. Believing that this phraseology is unsatisfactory, the

approximate diameter of an equivalent spherical mass is here used. A "7 mm. piece" of sodium would mean a mass whose general form if spherical would have an approximate diameter of 7 mm. As a matter of fact a piece of this dimension would ordinarily be designated as being the "size of a pea." A paper millimeter scale pasted to the table or wall will materially aid in estimating the various sizes.

Under the various subjoined heads suggestions are made regarding the general apparatus required.

Weights and measures. — The metric system of weights and measures alone is used. A meter stick and some cheap scales with a set of metric weights should be at hand. Sheets of paper of convenient size to place on the scale pans should be cut ready for use in weighing out solids. Corrosive solids should be weighed in previously tared watch-glasses. Liquids are best measured in the several sizes of graduated cylinders.

Glassware. — All glassware except test-tubes should be made of Jena glass. The use of this glassware is deemed of such importance that it is especially emphasized in the descriptions of many experiments. By thus including it in the specifications of apparatus, it must not be thought that other forms of glass will not serve the purpose, though the advantage always lies with the apparatus constructed with Jena glass.

It will be observed that all glassware required is of the nature ordinarily used in the laboratory. The Erlenmeyer form of flask is especially recommended, and in general is to be substituted for the older form, as it is much easier to clean, and the greater area of the bottom renders it more stable and results in a more even distribution of heat. Bulb-tubes are especially advantageous, but may in most

cases be replaced by short pieces of combustion-tube, though in this case if the substance to be acted upon is a liquid or an easily melted solid, it should be placed in a porcelain boat.

Test-tubes and ignition-tubes. — A rack should be filled with clean, dry test-tubes of different sizes, and an assortment of hard-glass test-tubes or ignition-tubes should be provided. A plate containing clean white sand is useful to lay ignition-tubes on, and to place under heated tubes to catch anything that might fall.

Kipp gas generators. — Though faulty in principle, the Kipp generator, or one of its various modifications, remains to-day the only portable gas generator for the lecture table. At least two generators are necessary, — one for hydrogen, and the other for carbon dioxide. A large quantity of dilute hydrochloric acid (1 : 1) should be made up ready for use in these generators. As a rule, the simpler and less expensive the form of Kipp used, the better. A tubulature in the lower chamber is desirable. Especial care should be exercised to have all joints and connections tight.

Batteries. — An open circuit battery, preferably of the "dry" form, and a "bichromate" battery of six cells are necessary. The expensive windlass form of bichromate battery may be replaced by simpler though less convenient forms at a much lower cost. The zincs should be amalgamated. An excellent battery solution is made as follows : —

600 g. potassium dichromate.
1000 cc. concentrated sulphuric acid.
4800 cc. water.

Splinters, tapers, and candles. — Splinters of wood are used frequently in testing for oxygen, and should accordingly be of a material that will retain a spark for some time. The

best splinters for this purpose are made from cigar-box wood. A supply should always be kept in a place where they cannot become wet. An assortment of candles consisting of short pieces of the ordinary household size, and especially the small candles so often used for decorative purposes, should be at hand. The latter will be found particularly convenient, as they can be readily thrust into a small-necked vessel. Suitable wires and supports for lowering the candles into a vessel or thrusting them up into an inverted jar are desirable.

Asbestos paper. — The free use of this paper, which should be about the thickness of ordinary blotting-paper, is especially recommended. As a protection to the table, as a heat distributor under a flask or beaker to be heated, and as a valuable accessory in many experiments, it is almost indispensable on the lecture table.

Compressed gases. — Cylinders of compressed or liquefied gases, if not of an unwieldy size, are an invaluable adjunct to a lecture table. Their high price prohibits their ever being universally used, but cylinders of liquefied carbon dioxide are found in many drug stores and confectionery shops. Liquefied nitrous oxide is used by many dentists, and may be borrowed, the gas used being determined by the loss in weight. Liquefied anhydrous ammonia is an essential in most refrigerating plants where a partially filled cylinder may often be borrowed. Compressed oxygen is of such importance that it has received special treatment in the text. All of these gases, together with hydrogen and sulphur dioxide, may be obtained in compressed or liquefied form from the regular dealers in chemical supplies. Where it is possible to obtain them near home, however, the expense of the express or freight, as well as rental on the cylinders, is saved.

Shields, gauntlets, and eyeglasses. — Experiments of a dangerously explosive nature are not to be recommended to the inexperienced. It is true, however, that experiments with explosive mixtures can be made on a small scale with practically no danger to experimenter or observers, by using small quantities of material and placing the apparatus between glass screens. In this way all flying particles of glass or chemicals are confined to the area between the shields, and no harm can possibly come. The simplest screen is represented in Fig. 50, p. 102, and can be cheaply constructed by any carpenter. Two such screens will serve as a protection in nearly all experiments, though in a few cases four may be used to advantage. One screen is placed between the apparatus and the audience, and the other between the experimenter and the apparatus. This leaves two danger zones lengthwise of the table, but if no inflammable materials are carelessly exposed on the table, no danger need be feared.

For many experiments a protection to the hand is indispensable. The driving glove or gauntlet (Fig. 7, p. 19) is especially well adapted to this work, as the coat sleeve may be thrust into the sleeve of the gauntlet, and thereby the possibility of bits flying up the open sleeve may be avoided.

In experiments where there is an evolution of intense light it is advisable to cover the eyes with colored glasses. Glasses of smoked or colored mica are furthermore useful as a protection in many operations of an explosive nature. With proper use of the glass screens, however, protection to the eyes except from the effect of bright light is seldom necessary.

Reagents and test papers. — A full set of the common qualitative reagents, together with the various test papers, such as red and blue litmus, turmeric and iodo-starch, as well as touch-paper, should be provided.

Precipitations may be made in glass cylinders or conical wine-glasses. If the precipitation is to be made in a hot solution, the test-tube on foot (Fig. 51, p. 103), made of thin glass which will stand heat, is the vessel best adapted. A supply of long and short glass rods whose ends have been rounded in the flame should be provided.

Aspirators, water pumps, and water blast. — The simplest form of aspirator is made by fitting a two-holed cork to a large bottle and inserting in one hole a glass tube extending to the bottom of the bottle, while a glass elbow is thrust into the second hole in the cork. By attaching a rubber tube to the long glass tube and starting the siphon thus formed, the water in the bottle will be siphoned off and air drawn in to take its place. If the bottle has a tubulature at the bottom, the siphon is dispensed with and water simply drawn off at the bottom, the air entering at the top.

A water suction pump will practically replace the aspirator, and in addition is invaluable in many other operations, such as rapid filtration, etc. The glass forms are less liable to corrosion, and if wound with several thicknesses of wire gauze, are not easily broken.

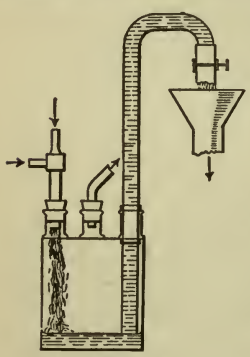

Fig. 1

A combined suction pump and water blast with suitable cocks for regulating the suction and the strength of blast is of great value. The expensive metallic forms may be replaced by the less convenient apparatus (Fig. 1) constructed by attaching a metal or glass filter-pump to one neck of a three-necked one-liter Wolff bottle. The middle neck is fitted with a rubber stopper and a glass elbow,

through which the blast of air is discharged. The third neck carries a cork through which a large glass tube extending to the bottom of the Wolff bottle is thrust. The upper part of the glass tube, which must be about twice as high as the Wolff bottle, is bent downward in the form of a U, and its opening so directed as to deliver the water into a sink or overflow. On allowing water to flow through the filter-pump, the air is drawn in, and the air and the water delivered into the bottle, the water collecting in the bottom of the bottle. If the air exit tube in the middle neck is not opened, the air is compressed sufficiently to force the water out of the bottle through the tube in the third neck. Soon the water will have been so far removed that water mixed with air is forced over. By opening the air-blast tube, air may be withdrawn at the rate desired, provided the level of the water in the bottle does not rise enough to flow out of the air-tube. With a good strong filter-pump sufficient air may be obtained to operate a blast-lamp.

Pneumatic troughs. — The pneumatic trough is indispensable in experimenting with many gases. When only small quantities of a gas are to be collected, a glass crystallizing dish is used, as the operation is then entirely visible. When larger quantities are to be operated with, a zinc, galvanized iron or copper trough is used. The metal should be well coated with asphalt varnish to resist the action of acids. Sinks and troughs set in the lecture table are a great convenience, but by no means indispensable.

OXYGEN

OXYGEN

PREPARATION

1. From mercuric oxide. — Owing to its historical interest, this experiment is almost invariably used in introducing the subject of oxygen.

A one-centimeter layer of dry mercuric oxide is placed in a hard-glass test-tube, and heated in a Bunsen burner pro-

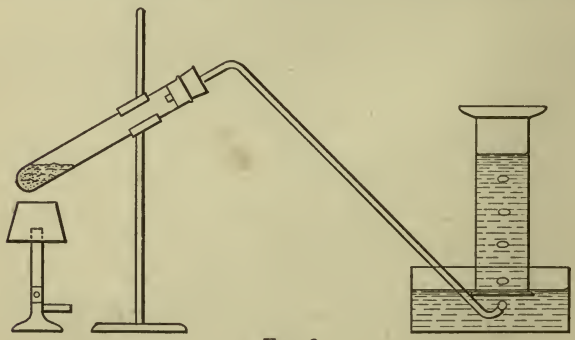

FIG. 2

vided with a chimney. The tube is clamped in an inclined position nearly horizontal, and the burner with its chimney so arranged (Fig. 2) that only the mercuric oxide is heated, that portion of the test-tube above the oxide remaining as cool as possible.

By using a somewhat larger portion of mercuric oxide, and fitting a cork with a delivery-tube into the mouth of the test-tube, sufficient oxygen may be obtained to fill several small jars at the pneumatic trough.

$$2\,HgO = 2\,Hg + O_2.$$

Bunsen burner and chimney; crystallizing dish and cylinders; HgO.

2. From silver oxide. — Another compound of oxygen that is easily decomposed by heat is silver oxide. The decomposition is attended by a characteristic change in color from the brown of the oxide to the silver-white of the metal.

The oxygen is evolved in such considerable quantities that when the test-tube is fitted with a delivery-tube, several small jars of oxygen may be collected at the pneumatic trough. The change in color above referred to especially recommends the method for lecture use, as the metallic silver is easily seen at a distance. If the heat is not high enough to fuse the glass, metallic silver in a semi-porous lump may be shaken out of the test-tube and allowed to fall on a plate, thus further showing its metallic character.

On dissolving the residue in nitric acid and precipitating with an excess of sodium hydroxide, hydrated silver oxide is obtained, which, on being filtered and dried, yields the original product ready for a repetition of the experiment.

$$2\,Ag_2O = 4\,Ag + O_2.$$

Test-tube and delivery-tube; several 50 cc. cylinders; porcelain plate; dry Ag_2O.

3. By heating potassium chlorate in the presence of small quantities of other oxides. — The catalytic action of manganese dioxide and ferric oxide on molten potassium chlorate, causing the rapid evolution of oxygen, illustrates the advan-

tage of using these oxides with potassium chlorate in the preparation of oxygen.

Potassium chlorate is melted at a low temperature in a hard-glass tube, and the absence of oxygen shown by a glowing taper. The salt must not be heated much above its melting point. On removing the lamp and immediately dropping in a small pinch of powdered manganese dioxide, a vigorous evolution of oxygen ensues. The experiment may be repeated with another tube of molten potassium chlorate, using ferric oxide instead of manganese dioxide. The demonstration of the unchanged nature of the oxides is not adapted to lecture-table experimentation.

$KClO_3$; MnO_2; Fe_2O_3.

4. By heating a mixture of potassium chlorate and manganese dioxide. — As was seen in the preceding experiment, the presence of manganese dioxide influences the liberation of oxygen from potassium chlorate in two ways: first, the temperature at which oxygen is evolved is much lower when a mixture of the two compounds is heated, than when the potassium chlorate is heated by itself; second, the evolution of oxygen from the mixture is much more regular. Accordingly, in the preparation of large quantities of oxygen, the mixture is almost invariably used. The two ingredients must be pulverized and thoroughly dried.

Twenty grams of each are intimately mixed and placed in a 250 cc. Jena glass Erlenmeyer flask fitted with a cork and a rather wide glass elbow, and clamped on a ring-stand. To the elbow is attached a rubber tube fitted with a glass elbow at the other end, which permits of a free movement of the tube in the pneumatic trough. On gradually increasing the heat, a rapid, though steady, evolution of oxygen ensues. The gas may be collected in several glass cylinders for special experiments, or may be transferred to one of the

many forms of gas holders. At the end of the reaction, the tube must be removed from the pneumatic trough to prevent the back suction of the water. The purity of the manganese dioxide is of considerable importance, as an admixture of carbonaceous matter of any nature is likely to cause an explosion. It is advisable to test a small portion of the mixture by heating it in a test-tube. If glass other than Jena is used, the flask should be protected in heating by a piece of asbestos paper.

$$2\,KClO_3 = 2\,KCl + 3\,O_2.$$

250 cc. Erlenmeyer flask (Jena) ; 1-holed rubber cork and two wide (7 mm.) glass elbows ; dry MnO_2 ; $KClO_3$.

5. From sodium peroxide. — Water decomposes sodium peroxide with the formation of sodium hydroxide and oxygen.

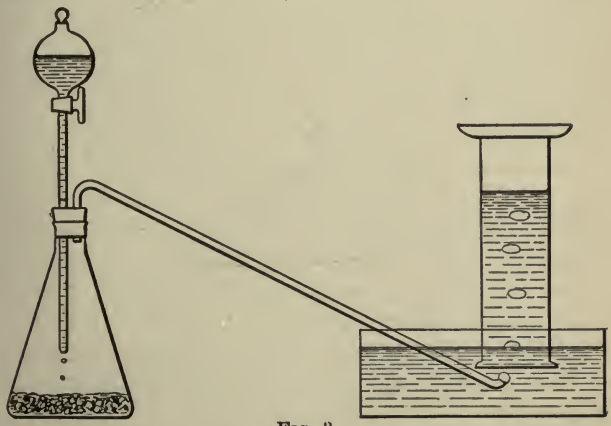

FIG. 3

If water in a dropping-funnel is allowed to fall, drop by drop, on 5 g. of dry sodium peroxide in a dry 100 cc. Erlenmeyer flask fitted with a delivery-tube (Fig 3), a

steady evolution of pure oxygen is secured. The yield of oxygen is very good, 6.25 g. of the peroxide giving 1 l. of the gas. As the volume of the ingredients is small, a small flask is used, and consequently there is no great volume of air to displace before beginning to collect the product.

The advantages of this method of preparing oxygen are numerous. Peroxide of sodium, while but recently introduced into the market, is obtainable at a very reasonable price, ranging at this date (1901) from 75 cents per pound in one-pound packages to 40 cents per pound in large quantities. At the higher price the cost of 1 l. of oxygen prepared in this manner is but a trifle over 1 cent; therefore the method is in no sense expensive. However, owing to its tendency to deteriorate in the air, sodium peroxide must be preserved in tightly stoppered bottles, preferably (in case of long standing) sealed with a little melted paraffin. The apparatus necessary for the operation is extremely simple, and as no heat is necessary, breakage seldom occurs.

The chemistry of the reaction is certainly no more complex to the elementary student's mind than that of the potassium chlorate reaction, especially when the introduction of manganese dioxide must be explained to the class.

The regulation of the flow of oxygen is nearly perfect, as each drop of water generates a certain amount of oxygen; thus a few centimeters or an almost unlimited volume per minute can be obtained by simple regulation of water supply. Only a small quantity of water, however, is necessary to decompose the peroxide.

$$2\,Na_2O_2 + 2\,H_2O = 4\,NaOH + O_2.$$

100 cc. Erlenmeyer flask; 50 cc. dropping-funnel; glass elbow; rubber stopper; Na_2O_2.

6. By the action of the chlorophyl of green leaves. — The exhalation of oxygen by green leaves under the influence of sunlight may be readily shown by filling a 2 l. flask containing green leaves (not too closely packed), with water through which carbon dioxide has been allowed to bubble (Fig. 4). A large funnel is passed through a hole in the cork, which is firmly pressed down into the flask in such a manner that the carbon dioxide water will rise, thereby expelling the air in the stem of the funnel. The funnel is two-thirds filled with water, and a small cylinder filled with water is inverted with its mouth directly over the stem of the funnel. The whole apparatus is placed in bright sunlight, and in a few hours sufficient oxygen will have risen and displaced the water in the small cylinder to give a good test. The apparatus may be left, and the oxygen tested at the next exercise. If the carbon dioxide water is not too strongly charged with the gas, it will be unnecessary to remove the trace of the carbon dioxide from the oxygen before testing.

FIG. 4

Apparatus (Fig. 4) ; green leaves ; carbon dioxide water.

7. From the electrolysis of copper sulphate. — In the electrolysis of copper sulphate, where but one of the final products of electrical decomposition is gaseous, that product, oxygen, may be isolated and tested. (Compare with the electrical decomposition of water, sodium sulphate solution, etc., where two gaseous products are formed.)

The glass bottle of the electrolytic apparatus (Fig. 5) is completely filled with a saturated solution of copper sulphate, and the cork inserted in such a manner as to drive out all the air and fill the delivery-tube with the solution. If a current from four cells of a bichromate battery is passed through the apparatus, a steady stream of oxygen will be delivered. One of the platinum electrodes, the negative one, will become coated almost instantly with metallic copper; the other, from which the bubbles of gas rise, will retain its original color.

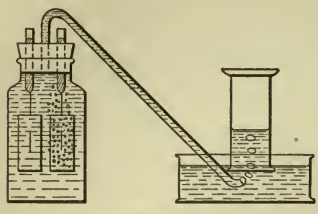

FIG. 5

Pieces of cardboard with the signs + and − are placed in a proper position to show the electrical connection.

$$2\,CuSO_4 + 2\,H_2O = 2\,Cu + 2\,H_2SO_4 + O_2.$$

Electrolytic apparatus (Fig. 5) ; battery ; saturated $CuSO_4$ solution.

8. Compressed oxygen. — No gas is used as frequently as oxygen on the lecture table. Uniting, as it does, with almost every element, in most instances with brilliancy, it is an essential factor in chemical experimenting. The method of obtaining this gas, given in Ex. 5, is the most advantageous of all methods involving the preparation of the gas, but, as a ready supply of oxygen for lecture table as well as for other laboratory purposes, there is nothing so satisfactory and reliable as a cylinder of compressed oxygen such as is obtainable in the market.

Oxygen as ordinarily used in the laboratory is delivered from a gasometer either of metal, or of glass with metal connections. These gasometers, owing to the corrosive action

of acids and fumes in the laboratory, soon begin to leak, and are repaired with great difficulty. Furthermore, their chief use is to store either air or oxygen, as the Kipp form of generator furnishes a steady, constant supply of many gases such as hydrogen, carbon dioxide, hydrogen sulphide, hydrochloric acid gas, etc. Since by means of the water-blast a steady current of air may be readily obtained, the gasometer is necessary only as a holder of oxygen. The cost of a Pepys or Mitscherlich gasometer is very considerable, and for a much less sum a steel cylinder holding 10 cubic feet of oxygen may be obtained. By simply opening the valve in the top of the cylinder, the gas is allowed to flow out at any desired rapidity from one bubble a second upward.

A so-called "commercial oxygen," which has slight traces of carbon dioxide and nitrogen as impurities, may be obtained from the S. S. White Dental Mfg. Co., Princess Bay, N.Y., at the very reasonable rate of ten cents per cubic foot. (One cubic foot equals 28.3 l.) This gas is sufficiently pure to be used in elementary organic analysis after being freed from the small amount of carbon dioxide. A less pure, though for experimental purposes equally good, oxygen may be obtained from the dealers in calcium light supplies.

A pressure regulator, while by no means indispensable, is of great advantage in using a cylinder of compressed gas. Owing to its cost, however, its use is not likely to be universal, and recourse must be had to a simpler device which, with a little care, gives equally satisfactory results. The stem of a long T-tube is thrust through a two-holed rubber stopper which is fitted into the mouth of a 50 cc. cylinder (Fig. 6). The tube should extend nearly to the bottom of the cylinder, which should be covered with a 2 cm. layer of mercury. One arm of the T is connected with

FIG. 6

the oxygen cylinder, and the other arm is connected with the apparatus to be filled by means of a rubber tube provided with a screw pinch-cock.

When the pinch-cock is open, the oxygen flows directly from the cylinder through the T into the vessel. If the pinch-cock is closed, the oxygen flows down through the stem of the T and bubbles through the mercury.

In many cylinders the rate of the issuing gas may be very accurately controlled by the operation of the valve in the cylinder. In some, however, it is difficult to open the valve slowly enough to prevent a sudden rush of gas, which might prove a serious difficulty in many operations. With such cylinders the gas is conducted through the T-tube, one arm of which is closed. The rushing gas bubbles through the mercury and, by closing the valve on the cylinder, the current may be so regulated that the gas slowly bubbles through the mercury. By means of the screw pinch-cock oxygen may now be withdrawn at any rate desired. It is always possible so to regulate the pinch-cock and the valve on the cylinder that no gas escapes through the mercury.

This mercury safety-bottle serves the additional purpose of furnishing a vent for the gas in case the tubes of the apparatus used for the experiment become clogged. Soon the pressure would rise sufficiently to cause the gas to bubble through the mercury, where from its sound it would immediately attract attention. It is unnecessary, however, to use the regulator if the cylinder is provided with easily controlled valves, as the danger resulting from the stoppage of tubes is very slight.

The gas may be collected in cylinders over water or, owing to the greater density of oxygen, it may be collected more conveniently by displacement in dry jars. A glass tube leading to the bottom of the jar to be filled is connected with a rubber tube to a cylinder. On opening the valve the

gas is slowly allowed to enter the jar and, as it rises, push out the air at the top. A glass plate is slipped over the mouth of the jar, and a glowing splinter held at the opening beside the glass tube. When the jar is filled, the glowing taper will be ignited. The gas should not be shut off until the tube is withdrawn. The moment the end of the glass tube is drawn out of the jar, the glass plate is slipped on to seal the mouth of the cylinder. One great difficulty in filling jars by this method is the fact that the gas is often allowed to flow at too rapid a rate, thus causing air currents inside the cylinder and a consequent test for oxygen before the jar is really filled. It is well to dip the end of the delivery-tube into water for an instant to see how rapid the flow of gas is, before lowering it into the jar to be filled. The rate of flow may also be determined by inserting a gas washing-bottle between the cylinder and the jar.

Some manufacturers give the net weight of the cylinder as well as the weight of the oxygen it contains. In such cases, by keeping a record of the weight of the cylinder, it is easy to determine how much oxygen remains at any given time.

PROPERTIES

9. Combustion of wood. — The increased brilliancy of combustion, as well as the rekindling of a glowing taper, may be shown by thrusting a splinter, the end of which is glowing, into a cylinder of oxygen. The experiment may be repeated a number of times, and, if care is taken not to thrust the taper too deeply into the jar, and not to let it remain there too long to consume the oxygen, it will be seen that at each repetition the splinter must be thrust a little deeper to effect rekindling.

It is somewhat difficult to get a wood that will retain a

spark, though splinters made from cigar-box wood give satisfactory results. It is best, in extinguishing the flame from the burning taper, not to blow it out, but to shake it out by a sudden twist of the hand, as in this way the spark is more likely to be retained.

10. Combustion of a candle. — Owing to the fact that there are considerable quantities of gas generated in the combustion of a candle, its burning in oxygen presents a condition slightly different from that where a splinter is used. If the candle is lowered immediately after being blown out, the gas generated by the still hot wick will cause a slight explosion when the wick is rekindled, especially noticeable if the oxygen is in a rather tall jar.

Small candle on wire; jar of oxygen.

11. Combustion of charcoal. — Charcoal, when burning in the air, gives no flame, but simply glows. In oxygen no flame is obtained, though the glowing may approach vivid incandescence.

A glass cylinder of 1 l. capacity is filled with oxygen and covered with a glass plate. A 3 cm. piece of charcoal, preferably of bark, is supported in a loop on one end of a stout copper or iron wire some 40 cm. in length. On heating the charcoal in a Bunsen burner until it begins to glow and then quickly introducing it into the jar of oxygen, the increased brilliancy of combustion is very marked. If the copper or iron wire is too fine, it is likely to melt or burn and allow the charcoal to fall to the bottom of the jar. When the charcoal ceases to glow, the wire is withdrawn, and some clear limewater poured into the jar, the glass plate replaced, and the cylinder shaken, after which the contents may be poured into a glass. The

limewater is cloudy, with a white precipitate of calcium carbonate.

$$C + O_2 = CO_2.$$

3 cm. piece of charcoal on wire; 1 l. cylinder of oxygen; limewater.

12. Combustion of powdered charcoal. — (*a*) While the combustion of a solid lump of charcoal gave no appreciable flame, the combustion of hot finely powdered charcoal in oxygen is attended with a flame of intense brilliancy. An ordinary iron saucer, such as is used for a sand bath, is one-third filled with finely powdered charcoal (willow charcoal is the best) and heated until the charcoal just begins to glow in spots. A grocer's sugar funnel or an ordinary glass funnel, cut so as to make the frustum of a cone, is quickly slipped into the mouth of a cylinder of oxygen of not less than 1 l. capacity. The red-hot charcoal is immediately poured from the iron dish into the funnel, care being taken to use the crucible tongs and to protect the hands with gauntlets. The face should not be held too near the jar, as the flame reaches a considerable height. When the hot charcoal falls through the oxygen, the combustion is of almost explosive violence and dazzling brilliancy (Fig. 7).

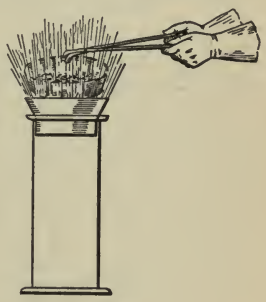

FIG. 7

Large wide-mouthed bottle or jar of oxygen (Fig. 7); grocer's tin sugar funnel; powdered willow charcoal; iron dish; crucible tongs; gauntlets.

(*b*) A stream of oxygen, when allowed to play on glowing, powdered charcoal, produces a most vivid combustion.

Finely powdered willow charcoal is placed in a small iron

dish (sand bath) and heated until the surface begins to glow. The flame is then removed, and a stream of oxygen is passed through a long glass tube with a bend at the end and allowed to play over the surface of the glowing charcoal. The combustion becomes very vivid, and if the stream of oxygen is of sufficient force to blow the dust out of the dish, the brilliancy of the combustion is almost blinding (Fig. 8).

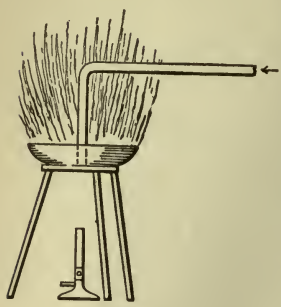

FIG. 8

Iron dish (sand bath); powdered willow charcoal; current of O.

13. Combustion of sulphur. — A porcelain crucible about the size of a thimble is so suspended in a loop of stout iron wire that it may be lowered into a jar of oxygen. The crucible is three-fourths filled with small bits of lump sulphur which are heated to boiling. On lowering the burning sulphur into a 500 cc. jar of oxygen, the combustion is continued with great brilliancy, resulting in the production of a blue flame. That the product of combustion differs materially from oxygen may be shown either by bleaching moistened blue litmus paper or by thrusting a piece of filter-paper moistened with potassium dichromate solution into the jar. In the latter case the red color of the dichromate is turned deep green by reduction.

$$S + O_2 = SO_2.$$

Small porcelain crucible and stout iron wire; 500 cc. jar of oxygen; lump sulphur; $K_2Cr_2O_7$ solution.

14. Combustion of phosphorus. — (*a*) The brilliancy of the combustion of phosphorus in oxygen is so great that this experiment has frequently been termed the "mock sun."

A 5 mm. piece of phosphorus, which has been carefully dried between filter-paper,[1] is placed in a small deflagrating spoon and a short piece of cotton twine laid beside it in such a manner that when a spark is started at the end of the twine it will serve as tinder to light the phosphorus on its introduction into the oxygen. The oxygen is held in a large flask or bottle, the bottom of which is covered with a little water. The deflagrating-spoon is previously arranged to allow the phosphorus to burn near the bottom of the vessel. On lowering the deflagrating-spoon into the jar, the glowing bit of twine bursts into a flame, which is immediately communicated to the phosphorus, and the combustion continues with dazzling brilliancy. White clouds of phosphorus pentoxide fill the whole jar, and by reflection of the light make the interior of the vessel luminous. If the flask was filled with oxygen by the displacement of air and the interior is dry, the phosphorus pentoxide will settle as a white hygroscopic powder all over the interior of the flask. In this case, however, it is advisable to place a layer of sand in the bottom of the vessel. A very small flame should be used to start a spark on the twine, and care taken to prevent a premature ignition of the phosphorus.

$$P_4 + 5\ O_2 = 2\ P_2O_5.$$

Large flask or jar of oxygen; deflagrating-spoon; cotton twine; P.

(*b*) The diminution in volume of the gas by the combustion of phosphorus in oxygen is shown by using an apparatus similar to that of Fig. 78, p. 182. The liter bell-jar used in this experiment should be tubulated. A crucible cover containing .5 g. of red phosphorus is placed on a large cork floating on the surface of water in a pneumatic trough. The bell-jar is then placed over the float, and by removing the stopper oxygen is conducted

[1] For precautions in handling phosphorus, see p. 232.

into the jar, thereby driving out the air. When all the air has been removed and a good test for oxygen obtained at the mouth of the bell-jar, a bit of string or paper which has previously been ignited is dropped upon the crucible lid, which has been brought under the opening in the bell-jar by means of a long wire. The cork is then immediately inserted. As the phosphorus burns, the water rises in the jar, and it will thus be seen that a considerable quantity of oxygen will be consumed.

Cork ; crucible lid ; tubulated liter bell-jar ; string ; red P.

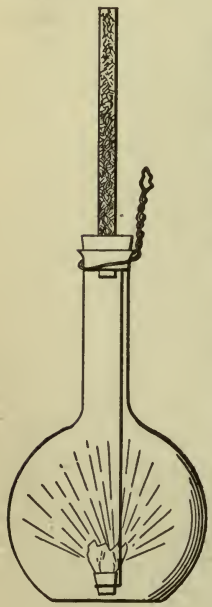

Fig. 9

15. Quantitative combustion of phosphorus in oxygen. — Phosphorus burning in oxygen forms phosphorus pentoxide, a white powder, which weighs considerably more than the phosphorus used. The increase in weight is shown by burning the phosphorus in a confined volume of oxygen in such a manner that the product of combustion is retained.

A dry two-liter flask is filled with oxygen and fitted with a one-holed rubber stopper carrying a 20 cm. length of glass tubing, 7 mm. in diameter, which is loosely filled with coarse glass wool or asbestos (Fig. 9). A stout copper wire is fastened to the stopper with the lower end, which is provided with a loop in which a small crucible is placed, near the bottom of the flask. The crucible should not contain more than 1 or 2 cc., and should be about two-thirds filled with red phosphorus. One end of a 2 cm. length of string is inserted in the phosphorus in such a man-

ner that the other end may be ignited in the air. The system is then brought into equilibrium on the lecture-balance. The stopper is removed, care being taken not to disturb the crucible or contents, the string ignited, and the stopper again thrust into the flask, where the string communicates the fire to the phosphorus, which in turn burns with a bright flame. The glass wool prevents the exit of the phosphorus pentoxide, which remains as a dry white powder covering the bottom and a portion of the sides of the flask. After a few minutes the increase in weight of the system, amounting to about .5 g., will be very apparent.

Lecture-balance; cork and tube with glass wool; copper wire; small crucible; string; O supply; red P.

16. Combustion of "steel wool" in oxygen. — Of the numerous forms of finely divided iron, that best adapted for combustion in oxygen is the so-called "steel wool" or steel fibre, which, as the name indicates, is a mass of fine shreds of steel. This product is used extensively for polishing hardwood, and is accordingly obtainable from most dealers in hardware or painters' supplies. A German product, which is sold only in pound packages, is but very little, if any, better than the American product, which can be obtained in quantities as small as desired.

A bit of the wool heated in the air will continue to burn for a few moments.

It is in pure oxygen, however, that the brilliancy of the combustion is best observed. A tuft of the wool is fastened on the end of a stout iron wire, heated in the Bunsen flame to incipient combustion, and then thrust into a jar of oxygen, on the bottom of which a layer of water, sand, or asbestos paper is provided. The wool burns with beautiful scintillations, the iron oxide falling to the bottom in fused globules.

Jar of O with H_2O, sand, or asbestos; "steel wool."

17. Combustion of aluminium. — Aluminium in the form of thin leaf is as combustible in oxygen as magnesium or any of the other finely divided metals, while in air it is not easily ignited.

Three or four leaves of aluminium are loosely bound with a stout iron wire and lowered into a jar of oxygen. A centimeter piece of twine fastened to the loop of the iron wire and ignited an instant before being thrust into the jar serves to kindle the aluminium leaf. The combustion is instantaneous and accompanied by a bright flash. If the iron wire is too fine, it also will burn in the oxygen.

Aluminium leaf; 300 cc. cylinder of O.

18. Combustion of finely divided magnesium, aluminium, zinc, or iron in oxygen. — The brilliancy of the combustion of these metals in oxygen is shown by causing each of them to be blown lengthwise through the Bunsen flame by a sudden puff of oxygen. Each powder is placed in the bend of a glass elbow, of 5 or 6 mm. internal diameter, in such a way that it does not quite obstruct the passage of the gas through the tube. A very gentle stream of oxygen is allowed to flow through the elbow over the metal till all the air is driven out. On directing the open end of the elbow toward the Bunsen flame, and suddenly admitting a puff of oxygen or air, the greater portion of the powdered metal will be shot across or through the flame and there be burnt with a blinding flash. The elbow should be so directed that the powder will be blown through the length of the flame. As considerable care is necessary to secure the proper introduction of the metal into the glass elbow, it is advisable to have prepared four elbows, all properly filled with the respective powders.

Four glass elbows (6 mm. diameter); Mg, Al, and Fe powders; Zn dust.

19. Explosion of a mixture of powdered charcoal and oxygen. — Oxygen ladened with charcoal dust forms an explosive mixture which varies in violence with the amount of dust suspended in the gas. Variations in the rapidity of combustion may be easily studied by means of the following experiment: —

A 5 mm. layer of finely powdered willow charcoal is placed in the bottom of a dry 500 cc. cylinder. A stream of oxygen is directed through a glass tube which extends to the bottom of the glass cylinder and is bent at right angles 3 cm. above the mouth of the cylinder (Fig. 10) in such a manner that the current of gas stirs up the charcoal powder in the bottom of the cylinder and fills it with a dust-ladened atmosphere of oxygen. The oxygen is then cut off and a flame instantly applied at the mouth of the cylinder. By varying the rapidity of the current of oxygen, and consequently the proportion of the dust in the gas, the flame may be made to travel down the cylinder with varying degrees of velocity, affording an interesting study of the rate of propagation of an explosion. This and the following experiment are striking illustrations of the explosion of a solid in a gas.

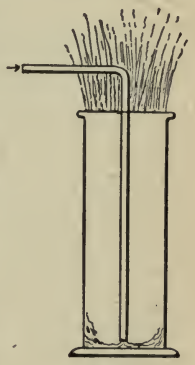

FIG. 10

500 cc. cylinder; powdered willow charcoal; current of O.

20. Explosive combustion of powdered iron, aluminium, or zinc in oxygen. — An atmosphere of oxygen ladened with the dust of iron, aluminium, or zinc, possesses explosive properties.

Oxygen is conducted through a bent glass tube to the bottom of a glass cylinder, 3 cm. in diameter and 15 cm.

high. A 5 mm. layer of powdered iron or aluminium or zinc dust is placed on the bottom of the jar. The stream of oxygen is so directed as to blow the dust up into the cylinder and produce an atmosphere of oxygen ladened with the metallic dust. On applying a flame at the mouth of the cylinder the mixture explodes. The brilliancy of the combustion renders the use of colored glasses necessary.

100 cc. glass cylinder ; colored glasses ; current of O ; powdered Fe, Al ; Zn dust.

21. Absorption of oxygen by potassium pyrogallate. — Oxygen is collected in a eudiometer (Fig. 11), and potassium pyrogallate solution is allowed to flow down through the stopcock into the gas. The absorption of the oxygen is almost immediate, the water rising to take the place of the absorbed gas.

FIG. 11

Potassium pyrogallate solution is best made by dissolving 10 g. of pyrogallic acid in a solution of 100 g. of stick potassium hydroxide in 500 cc. of water. Made with these proportions the solution absorbs oxygen readily and, if tightly corked, keeps well.

Eudiometer (Fig. 11) ; pyrogallic acid ; stick KOH ; O.

22. Combustion of zinc in air. — At the ordinary temperature it is extremely difficult to ignite zinc, though, when at or near its boiling point, combination with the oxygen of the air takes place easily.

Three or four grams of zinc (not granulated) are heated in a small porcelain crucible with a blast-lamp. After a few minutes the zinc boils and catches fire on the surface. The zinc oxide formed collects in white flocks around the edge of the crucible, which, on cutting off the blast, ascend with the

hot current of air in white clouds. On cooling, the flocculent nature of the zinc oxide may be observed.

$$2\ Zn + O_2 = 2\ ZnO.$$

Crucible ; blast-lamp ; Zn.

23. Combustion of iron in air (quantitative experiment). — A small magnet to which a considerable quantity of iron powder is hanging is suspended from one arm of a lecture-balance which is brought into equilibrium. The iron is then ignited by gently playing a Bunsen flame upon it, care being taken not to disturb the balance. In a few moments sufficient oxygen will have combined with the iron to produce a very considerable change in the equilibrium of the balance.

A much larger amount of iron, consequently showing a greater deflection, can be burned by placing the iron powder on a square of sheet asbestos that has previously been ignited. The asbestos is placed on a square of wire gauze, and so laid on the balance pan as to prevent undue heating and consequent unsoldering of its supports. The balance is then brought into equilibrium, and the iron ignited by means of a small gas-jet. The combustion continues of itself, and soon a large deflection of the balance is noticeable.

Magnet ; lecture-balance ; ignited asbestos paper ; square of wire gauze ; iron powder.

24. Combustion of magnesium in air (quantitative experiment). — Magnesium powder burns quietly in the air, forming white magnesium oxide.

Five grams of magnesium powder are heaped on a 12 cm. square of previously ignited asbestos paper, which is in turn placed on the pan of a lecture-balance, with all the precautions of the preceding experiment. After bringing the

system to equilibrium, the magnesium is ignited with a match or gas-jet. The combustion soon proceeds through the whole mass, which increases in volume considerably, and a small portion of the magnesium oxide formed escapes as a white smoke. After the mass has become cool, approximately 2 g. will be required to restore the equilibrium.

Lecture-balance ; wire gauze ; asbestos paper ; Mg powder.

25. Increase in weight by the combustion of a candle in air. — In burning a candle, if provision is made to collect the products of combustion, *i.e.*, carbon dioxide and water, it will be found that they weigh more than the candle consumed.

FIG. 12

A cylindrical Argand lamp chimney, 21 cm. high, 5.5 cm. in diameter, is provided with a false, wire gauze bottom, as is shown in Fig. 12. A circular piece of rather coarse wire gauze is cut of such a size as to readily pass through the chimney. Three pieces of fine copper wire, 50 cm. long, are fastened at three equidistant points in the circumference of the gauze by tying a knot in the middle of each of the three strands. The gauze bottom is placed inside the chimney 6 cm. from one end, and fastened in this position, by bringing the wires up on the outside as well as the inside of the chimney and twisting the ends of each wire together at the top. One end of each strand is left long enough to twist all three together and form a suspension for the chimney. A few lumps of quicklime are placed on top of the wire gauze and the rest of the space nearly filled with stick

sodium hydroxide. A large cork is fitted to the lower part of the chimney. Several good-sized holes are made through the cork to allow for the draft, and the ring of a crucible lid is pressed into a slit in the centre of the cork, forming a pan on which a candle is placed. The whole apparatus is suspended on the arm of a lecture-balance and brought into equilibrium. The cork is removed, the candle lighted, and the cork quickly replaced. If the holes in the cork are of sufficient size, there will be a good draft and the candle will burn freely. The candle is of ordinary size and rather short, about 15 mm. high. Any wax dropping from the candle is caught in the crucible cover. The holes in the cork can be made by cutting V-shaped sections out of the edge. After burning a few minutes a very perceptible increase in weight will be obtained.

Argand lamp chimney; wire gauze; lecture-balance; Cu wire (fine); quicklime; stick NaOH.

26. Quantitative combustion of phosphorus in air. — The apparatus of Ex. 15 may be used to demonstrate the increase in weight of phosphorus burning in air.

It is better to substitute yellow for red phosphorus and to induce the ignition by a strip of touch-paper (p. 355). A 7 mm. piece of well-dried phosphorus is placed in the crucible on top of a piece of touch-paper which protrudes some 2 or 3 cm. above the top of the crucible. The apparatus is then carefully tared, as before, the cork removed, and the touch-paper ignited. The time required for the touch-paper to communicate the flame to the phosphorus is generally sufficient to permit the insertion of the cork and the adjusting of the balance.

The phosphorus pentoxide formed settles to the bottom and sides of the flask as a dry, white powder.

After the combustion is complete, and the system has

reached the room temperature, an increase in weight of .5 g. will be obtained.

Apparatus (Ex. 15.) ; O supply ; yellow P ; touch-paper.

27. Increase in weight by burning sulphur in the air. — If the sulphur dioxide formed by burning sulphur could be collected, its weight would be exactly twice that of the sulphur used. By means of a slight modification of the apparatus of Fig. 12, p. 28, the increase in weight by burning sulphur in the air may be easily demonstrated, though it is impossible to make the experiment so far quantitative as to show any mathematical relation between the weight of the sulphur and the weight of the product.

The Argand lamp chimney, prepared and filled with quicklime and sodium hydroxide, as described in Ex. 25, is provided with a perforated cork carrying a crucible cover, a small disk of asbestos paper being placed between the cork and the crucible cover. A few bits of sulphur are placed in the crucible lid, the cork inserted in the chimney, and the whole brought to equilibrium on the lecture-balance. The cork is then removed, the sulphur ignited, and the cork replaced in the chimney. It is advisable to let the sulphur burn in the air for a moment or two to insure its thorough ignition. The sulphur burned by so waiting interferes in no way with the experiment. A few minutes after the cork is replaced in the chimney a marked increase in weight is observed.

Lamp chimney filled with lime and NaOH ; cork with asbestos disk and crucible cover ; S.

28. Absorption of oxygen by rusting iron. — The absorption of oxygen from the air by rusting iron can be readily effected by inserting a wad of steel wool (Ex. 16) into a eudiometer tube containing 100 cc. of air, inverted over

a crystallizing dish of water (Fig. 13). The eudiometer should first be filled with water and then enough of the water allowed to flow out of the tube to confine about 100 cc. of air. The plug of steel wool is then thrust under water and inserted in the tube by means of a long copper wire.

The apparatus thus prepared is allowed to stand twenty-four hours, when it will be seen that the iron has rusted considerably and that the volume of the enclosed gas has materially diminished.

Eudiometer tube ; steel wool ; Cu wire.

FIG. 13

OZONE

FORMATION AND PREPARATION

29. By heating potassium chlorate or mercuric oxide. — If a small quantity of potassium chlorate or mercuric oxide is heated in a hard-glass tube until oxygen is evolved, a piece of moistened iodo-starch paper held at the mouth of the tube will be turned a deep blue.

$$KClO_3 = KCl + O_3. \quad [?]$$
$$3\ HgO = 3\ Hg + O_3. \quad [?]$$

$KClO_3$; HgO ; KI-starch paper.

30. From barium peroxide and sulphuric acid. — By acting on a peroxide with a strong acid, ozone is formed in very appreciable quantities.

Three or four grams of powdered barium peroxide are carefully and gradually shaken into a small beaker containing 10 cc. of concentrated sulphuric acid externally cooled by ice. The air immediately above the beaker is very rich in ozone and will give any of the usual tests.

In this experiment the process may be reversed by dropping the cooled sulphuric acid upon the barium peroxide in the bottom of the externally cooled beaker.

$$3\ BaO_2 + 3\ H_2SO_4 = 3\ BaSO_4 + 3\ H_2O + O_3.\ [?]$$

BaO_2; ice.

31. By the action of sulphuric acid on potassium permanganate. — As the action of concentrated sulphuric acid liberates chromic anhydride from potassium dichromate (Ex. 2, p. 405), in like manner permanganic anhydride is liberated from potassium permanganate.

Permanganic anhydride, owing to its unstable nature, immediately decomposes into manganese dioxide and ozone, the latter being obtained in considerable quantities. This reaction is very violent, and consequently must be performed on a small scale only. It may be safely carried out, however, by placing 1 g. of powdered potassium permanganate in each of six small beakers standing in a large crystallizing dish, the bottom of which is covered with a centimeter layer of water. The potassium permanganate in the beakers is moistened with a few drops of water.

Into one of the beakers 2 cc. of concentrated sulphuric acid are poured. A vigorous reaction takes place, accompanied by a smoke consisting chiefly of brown manganese dioxide. A pinch of sulphur flowers dropped into the beaker is instantly ignited.

The second beaker is likewise treated with 2 cc. of concentrated sulphuric acid, and a small piece of phosphorus thrown into it. The combustion is instantaneous.

Into the third beaker, after the addition of sulphuric acid, 1 cc. of alcohol is poured from a test-tube fastened to the end of a long stick. The combustion is of almost explosive violence.

A polished silver coin, held by tongs above the mixture in the fourth beaker, is rapidly turned black, giving the characteristic ozone test. Then a slow stream of illuminating gas is allowed to pass through a glass tube held at the mouth of the beaker. It is ignited, and, by pinching the rubber tube, the flame may be extinguished and relighted a number of times.

A glass rod is dipped in the mixture in the fifth beaker, and then touched to the wick of an alcohol lamp. The oxidizing mixture adhering to the glass rod lights the lamp.

Four cubic centimeters of ether are poured from a test-tube along the floor, and one end of the moistened strip touched with a glass rod dipped in the fresh mixture prepared in the sixth beaker. The ether is ignited, and the flame travels along the floor. As but a few cubic centimeters of ether are used, the combustion is of short duration and harmless. As the ether burns, it will be seen that the vapor has rolled out on either side of the moistened strip.

In all these experiments, protection is necessary from flying drops of concentrated sulphuric acid that might spurt from the beakers in such vigorous reactions. After use, each beaker should be removed with crucible tongs, and plunged into a dish of water kept at hand for that purpose.

$$2\ KMnO_4 + H_2SO_4 = K_2SO_4 + Mn_2O_7 + H_2O.$$

$$Mn_2O_7 = 2\ MnO_2 + O_3.$$

Six 50 cc. beakers ; large evaporating dish filled with water ; large crystallizing dish ; test-tube on a long stick ; pulverized $KMnO_4$; S flowers ; small piece of P ; alcohol and alcohol lamp ; ether.

32. By slow oxidization of phosphorus. — When moist phosphorus is allowed to remain in contact with air, it is slowly oxidized, and a portion of the oxygen of the air is converted to ozone.

A few sticks of freshly scraped phosphorus, about 4 cm. in length, are placed on the bottom of a 2 or 3 l. flask, and half covered with water. The mouth of the flask is then loosely closed with a watch-glass, and the whole allowed to stand at the room temperature. In a few minutes the air inside the flask will be found to be rich in ozone, which instantly turns blue a moistened piece of potassium iodide starch paper. As it is desirable to pass the ozonized air through certain tubes, the flask should be selected with a neck small enough to be fitted with a two-holed rubber stopper which has two glass elbows thrust through it, the end of one of the elbows extending 10 or 12 cm. below the bottom of the stopper. When water from the faucet is allowed to run slowly through the shorter elbow, it gradually fills the flask, driving the ozone out through the elbow whose end is nearest the bottom. As rubber rapidly destroys ozone, the ozonized air must be drawn from below the rubber stopper (Fig. 14). The formation of ozone in this manner is dependent on the temperature, and at 20° it is not uncommon to have very strongly ozonized air in the flask at the end of half an hour.

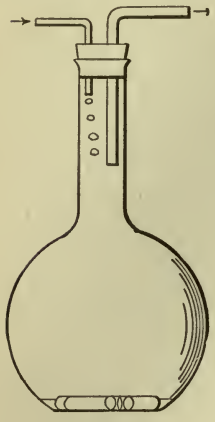

Fig. 14

A large bottle can be used instead of the flask.

Flask, 2 or more l. ; cork and tubes (Fig. 14) ; sticks of P 4 cm. long ; KI-starch paper.

33. From the slow combustion of ether in air. — When ether is burned in air under certain conditions, a product is formed giving the ozone reactions, and is commonly called ozone, though its nature is not thoroughly understood.

Two cubic centimeters of ether are poured into a 200 cc. cylinder and well shaken. When a platinum wire, which has been heated in a Bunsen flame and allowed to cool until the glow has disappeared, is lowered into the jar containing the mixture of ether vapor and air, a partial combustion takes place. A glass rod heated till the flame begins to be colored may be substituted for the platinum wire. The gaseous contents of the cylinder will give a decided ozone reaction when a strip of iodo-starch paper is introduced.

200 cc. cylinder; platinum wire; iodo-starch paper; ether.

PROPERTIES

34. Iodo-starch paper (potassium iodide and starch). — Ozone acts upon potassium iodide and liberates iodine. The free iodine immediately unites with the starch, forming the blue compound, and accordingly potassium iodide, in the presence of starch, gives an excellent test for ozone.

One gram of starch is suspended in 100 cc. of water which is brought to a boil. After the starch has been completely emulsified, .5 gram of potassium iodide is dissolved in the solution. Strips of filter or other bibulous paper are dipped in the solution and dried. When testing for ozone, a strip of the dried paper is moistened and held in the gas. The blue coloration immediately appears.

$$6\ KI + O_3 + 3\ H_2O = 6\ KOH + 3\ I_2.$$

35. Preparation of ozone paper (potassium iodide and red litmus). — In the experiment with iodo-starch paper, in which the free iodine liberated combines with the starch to form the blue compound, the potassium hydroxide formed can be utilized to turn red litmus paper blue, and thus serve as a delicate test for ozone.

Strips of blue litmus paper are dipped in a solution com-

posed of 100 cc. of water, 1 drop of sulphuric acid, and .5 g. of potassium iodide. Red litmus paper may be used and the sulphuric acid omitted. For preservation, the strips are dried, and require moistening before using. When the moist paper is held in the presence of ozone, the color rapidly turns blue.

36. Action on mercury. — Ozone acts on pure mercury, destroying its lustre and its mobility.

Two 100 cc. stoppered cylinders, each containing 5 cc. of pure dry mercury, are filled, the one with oxygen and the other with ozonized air, from the apparatus, Fig. 14. A strip of iodo-starch paper is held in the mouth of each cylinder, and while it is unacted upon by the oxygen, it will be immediately turned blue by the ozonized air. Each cylinder is then closed and vigorously shaken, when it will be found that the mercury in the oxygen cylinder is unacted upon, while the mercury in contact with the ozonized air will be tarnished and will stick to the walls of the vessel when shaken. That the ozone has been destroyed is shown by holding a strip of iodo-starch paper in the mouth of the cylinder. Its color will not be changed.

Two 100 cc. stoppered cylinders; ozonized air apparatus (Fig. 14); O supply; pure Hg.

37. Oxidation of lead sulphide by ozone. — The oxidizing action of ozone is shown by its conversion of the black sulphide of lead to the white sulphate. Lead sulphide is formed by moistening a strip of filter-paper with lead acetate and exposing it an instant to the fumes of hydrogen sulphide. On suspending the blackened paper in a jar of ozone, the black rapidly disappears.

38. Decomposition by copper oxide. — At the room temperature copper oxide decomposes the ozone molecule, forming ordinary oxygen.

A stream of ozonized air from the flask (Fig. 14) is passed through a 20 cm. piece of combustion tubing fitted with a cork at each end. A glass elbow dipping into a beaker containing potassium iodide starch solution is inserted in the cork at the farther end of the combustion tube. A slow stream of the ozonized air is now passed through the system, and its action on the potassium iodide starch mixture shown. A spiral of copper, made by winding some copper wire around a glass rod until the spiral has reached the length of 10 cm., is then heated until the surface of the copper is entirely oxidized. The cork containing the glass elbow is removed, and the copper oxide spiral, after becoming cold, is introduced into the combustion tube and the cork replaced. After allowing the ozonized air to pass through the system a few minutes, it will be seen that a fresh iodo-starch solution will not be acted upon by the gas bubbling through it. If the spiral is removed and the cork reinserted, the ozonized air will again change the color of the solution in the beaker.

To insure non-contamination of the iodo-starch solution, two corks carrying elbows should be provided, that they, as well as the beakers, may be changed.

Ozonized air in flask (Fig. 14, p. 34); 20 cm. length combustion tubing; two corks carrying glass elbows; two 50 cc. beakers; iodo-starch solution; copper spiral.

39. Decomposition by heat. — Any appreciable increase of temperature destroys the ozone molecule and consequently its property of setting free iodine from potassium iodide.

A gentle stream of ozonized air from the flask (Fig. 14, p. 34) is directed through a 5 mm. glass tube connected at one end with an elbow which dips into a small beaker containing potassium iodide-starch solution. On heating the glass tube with a Bunsen burner, the ozone is destroyed

and a fresh beaker of potassium iodide-starch solution is unacted upon by the gas that bubbles through. A fresh elbow must be connected to the tube.

All connections should be made with glass touching glass, the rubber only serving to hold the tubes together.

$$2\,O_3 = 3\,O_2.$$

Ozonized air in flask (Fig. 14, p. 34); two glass elbows (5 mm.); KI-starch solution.

40. Decomposition by rubber. — The decomposition of ozone by rubber and the consequent necessity of avoiding rubber connections in all ozone apparatus is shown by conducting a rapid stream of ozonized air through a 1.5 m. length of rubber tubing and testing the issuing gas with iodo-starch paper. The ozonized air is best obtained from the apparatus, Fig. 14, p. 34, and the water should flow into the flask at the rate of about 40 cc. in 10 seconds. The ozonized air should be tested just as it leaves the glass elbow, and then the rubber tube should be connected. After a few moments the gas issuing from the other end of the tube will be found to be free from ozone.

Ozonized air in apparatus (Fig. 14, p. 34); 1.5 m. length of rubber tubing.

HYDROGEN

HYDROGEN

PREPARATION

1. By the action of sodium on water. — Sodium reacts vigorously with water, liberating hydrogen and forming sodium hydroxide. The preparation of hydrogen by this reaction is only of theoretical interest, and should be carried out on a very small scale only.

A 5 mm. piece of sodium is cut from a large piece of the fresh metal whose crust has been removed, the whole operation being performed under petroleum, ether, or benzine. The small piece is then freed from petroleum by being carefully dried on a piece of filter-paper. It is necessary that the hands should be dry, as even small quantities of perspiration are liable to ignite the sodium and cause a bad burn. In case a bit of sodium becomes ignited, it can be extinguished readily by covering it with a handful of dry sand (water must never be used).

The sodium is now wrapped in a piece of thin sheet lead (the leaf lining to tea chests, obtainable at any grocer's, serves the purpose admirably), one end of the sodium being left uncovered. The lead should be pressed down on all sides closely, and the end opposite the exposed end tightly pinched together. On dropping the sodium prepared in this way into water in a crystallizing dish, the action is

very gentle, and hydrogen is steadily evolved (Fig. 15). By covering the sodium with an inverted jar full of water, the hydrogen may be collected. If the sodium is overlapped at its exposed end by the lead, a bubble of air often prevents the water from coming in contact with the sodium. The lead should be trimmed off just flush with the exposed surface of the sodium. When tea lead is used, it should be doubled, as one thickness is liable to be melted through by the intense heat of the reaction. Three or four pieces of sodium may be wrapped in separate bits of sheet lead, and when the gas evolution from one has ceased another may be thrown into the water and thus sufficient gas collected.

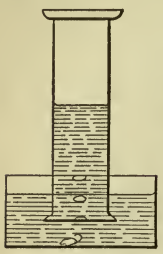

Fig. 15

The wire gauze baskets so often used in this experiment are liable to introduce a considerable quantity of air into the gas, with the possible formation of an explosive mixture. Furthermore, when a second piece of sodium is to be introduced in order to fill a jar with the gas, a second dry basket must be used. The introduction of bits of sodium on the end of a needle is not to be recommended, as the metal often becomes disengaged from the needle before it is under the jar, and rises to the surface of the water outside, and thereby causes annoyance.

Sodium, even under naphtha, oxidizes slowly, and while a number of pieces of sodium can be wrapped in lead and preserved in naphtha, the facility with which they can be prepared leaves very little to be gained by having a stock on hand. Freshly prepared, they will never fail to give a steady evolution of hydrogen.

$$2\,H_2O + 2\,Na = 2\,NaOH + H_2.$$

Metallic sodium (5 mm. pieces); tea lead; crystallizing dish and cylinder.

2. From the reduction of water vapor by iron. — Iron acts upon water at the ordinary temperatures very slowly. If, however, a current of steam is conducted over red-hot metallic iron, the reduction is very rapid, hydrogen being liberated.

A 60 cm. length of iron gas pipe is filled with nails and heated to redness in a small furnace or over a four-tube burner (Fig. 16). One end of the iron tube is connected with a steam generator and the other end fitted with a cork and a delivery-tube leading to the pneumatic trough. The steam generator consists of a 300 cc. Erlenmeyer flask fitted with a

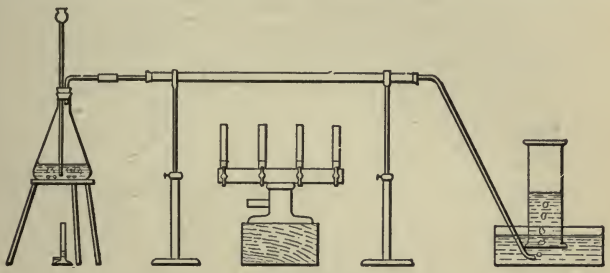

FIG. 16

two-holed rubber stopper, carrying a long thistle-tube and a glass elbow extending to the combustion-tube. The thistle-tube must extend to the bottom of the flask in which 100 cc. of water are brought to a boil. On passing a current of steam through the hot tube, a regular flow of hydrogen may be obtained at the pneumatic trough. Owing to the conduction of heat by the iron, care should be taken that the corks in the ends of the tube are protected from the heat. This can be accomplished either by having a rather long iron tube and heating only the middle portion, or by using red rubber stoppers, which stand a much higher temperature than ordinary cork or rubber stoppers. A still further pro-

tection may be secured by wrapping a thin sheet of asbestos around the corks before inserting them in the tube. By forcing the corks into the tube, a tight joint may be obtained.

60 cm. length gas pipe (1 cm. inter. diam.) ; iron nails ; 300 cc. Erlenmeyer flask ; long thistle-tube ; elbow ; 4-tube burner.

3. From the reduction of water vapor by zinc. — Finely divided zinc reduces water vapor, forming zinc oxide and liberating hydrogen. The temperature at which the reduction is effected by means of zinc is, however, very much lower than that required when using iron, and it is only necessary to heat the tube, which is of glass rather than of iron, a little above the boiling point of water. The apparatus is similar to that used in the preceding experiment. The combustion-tube is filled with zinc dust and gently heated with a four-tube burner. As the zinc becomes converted to zinc oxide, a marked color-change in the contents of the tube is noticed, and at times the rapidity of the reduction is such as to cause the zinc to glow.

$$Zn + H_2O = ZnO + H_2.$$

Glass combustion-tube ; apparatus of preceding experiment ; Zn dust.

4. From the reduction of water vapor by magnesium. — See Ex. 1, p. 371.

5. From the ignition of sodium hydroxide and iron powder. — One gram of powdered sodium hydroxide and 20 g. of iron powder are intimately mixed and heated in a hard-glass test-tube fitted with a cork and a delivery-tube, the upper part of the mixture being heated first. A rapid stream of hydrogen is obtained, and the gas may be collected at the pneumatic trough.

Powdered NaOH ; Fe powder.

6. By heating calcium hydroxide and zinc dust or iron powder. — To illustrate a technical process for the preparation of hydrogen, a mixture of equal parts by volume of zinc dust and dry calcium hydroxide is heated in a hard-glass test-tube, provided with a cork and a delivery-tube, the end of which dips into a pneumatic trough. On applying heat, the liberation of hydrogen is very regular, and the gas may be collected in bottles for testing. It is important that the calcium hydroxide be dry, as otherwise an excess of moisture is likely to collect on the tube and run back, causing it to break.

Iron powder may be substituted for the zinc dust in the above experiment with equally satisfactory results.

$$Ca(OH)_2 + Zn = CaO + ZnO + H_2.$$

Dry $Ca(OH)_2$; Zn dust; Fe powder.

7. From aluminium and sodium hydroxide solution. — Fifty cubic centimeters of dilute sodium hydroxide solution and a few grams of scrap aluminium are placed in a 300 cc. Erlenmeyer flask fitted with a thistle-tube and a delivery-tube. In the cold the action is at first very slow, soon warming up of itself and becoming very vigorous. As some time is required for the reaction to take place in the cold, it can be hastened by warming slightly. When once started the flame must be extinguished. A large quantity of pure hydrogen may be obtained in this manner from a very small weight of the metal.

$$6\ NaOH + 2\ Al = 2\ Na_3AlO_3 + 3\ H_2.$$

300 cc. flask; thistle-tube; delivery-tube; aluminium scrap.

8. From zinc and sulphuric or hydrochloric acid. — This method for the preparation of hydrogen is almost univer-

sally used in the laboratory, by reason of its economy and simplicity.

Owing to its great importance, large quantities of hydrogen are used on the lecture table, and it is necessary to have one or more forms of apparatus that will yield considerable quantities of the gas.

A ready supply of hydrogen may be obtained from the apparatus of the preceding experiment. Granulated zinc is placed in the flask, covered with water, and a few cubic centimeters of concentrated sulphuric acid are then added through the thistle-tube, which should extend to the bottom of the flask. After the addition of acid, the flask and its contents are gently shaken. Instead of adding concentrated acid to the water in the flask, the acid may be previously diluted, and the cold dilute acid added. In this case the reaction is not as rapid at first. Dilute hydrochloric acid (1 : 1) is also used to prepare hydrogen in this manner, this dilution of acid producing a rapid gas evolution.

On account of the large surface which is presented to the action of the acid, the so-called granulated or feathered zinc (Ex. 1, p. 377) is preferred for making hydrogen. The diffusibility of hydrogen renders it absolutely essential that all apparatus used in its preparation should be gas-tight. Ground glass joints, if any, should be well lubricated with vaseline; rubber stoppers should be crowded into place, and, if necessary, all connections with rubber tube should be bound with wire.

After the generation of the gas has begun, it is necessary to allow the gas to escape till the air in the apparatus has been completely expelled by the hydrogen, as otherwise considerable danger may result from the ignition of an explosive mixture of hydrogen and air if the issuing jet of hydrogen is lighted too soon. According to the amount of air space in the generator and the purifying apparatus, the gas is

allowed to escape into the air for some time, and then it is better to test it before applying a match directly to the issuing gas. A simple method of testing the gas is to allow it to bubble through a soap solution contained in an evaporating dish and, *after removing the delivery-tube*, to light the bubbles and see if they give an explosion. If not, it will be safe to ignite the gas. Another method is to lead the gas, by means of a rubber tube, to the bottom of an inverted test-tube, the lighter hydrogen rising and pushing down the air in the test-tube. The delivery-tube is slowly withdrawn and the thumb placed over the mouth of the test-tube, which is then carried to the Bunsen flame and ignited. When the gas is perfectly pure, it should burn quietly and give a flame of sufficient duration to serve in igniting the hydrogen issuing from the generator. On the danger of premature ignition of a hydrogen jet, see Exs. 29 and 30.

Commercial zinc ordinarily has sufficient impurity to give a rapid action with dilute acid. At times, however, the zinc may be so pure as to give a rather slow action. In such cases, use is made of the electrolytic action between zinc and copper or platinum to accelerate a liberation of the gas, and a few cubic centimeters of copper sulphate solution, or a few drops of platinic chloride solution, are added to the acid in the generator. The zinc becomes coated with a fine deposit of copper or platinum, and the increased gas evolution is very marked.

Hydrogen may be collected either by displacement of air or at the pneumatic trough, the second of these methods being used when a dry cylinder of the gas is not required. In collecting the gas by displacement (the operation on a small scale has been described above), it is essential that a rather rapid stream of gas be used, to prevent the diffusion of air into the vessel to be filled.

The gas is dried by passing it through a gas washing-

bottle containing concentrated sulphuric acid. When the issuing gas is to be lighted, it should be dried by passing through a U-tube containing calcium chloride, or pumice-stone drenched with sulphuric acid. In either case the gas must have a clear passageway through the drier, that the flame may not be affected by the irregular bubbling through a liquid.

If a special purification is necessary, the gas may be conducted through a gas washing-bottle containing acidulated potassium permanganate solution.

The Kipp generator furnishes, at the same time, the most convenient, as well as the most satisfactory, apparatus for maintaining a constant supply of hydrogen. The simpler form of apparatus (Fig. 17) consists practically of two pieces, the base being a glass bottle constricted in the middle, forming two chambers, the upper chamber being fitted with a side tubulature in which a stop-cock is inserted, and the lower chamber tubulated for the removal of spent acid. The upper chamber contains the zinc, and the lower chamber serves as a reservoir for gas generated in excess of that wanted for immediate use. In the neck of the upper chamber is fitted a long funnel, the lower end of which extends down through the constriction to the bottom of the vessel. The funnel is made in the shape of a large bulb and serves as an acid reservoir. To charge the apparatus, granulated zinc is introduced through the opening in the upper chamber, and the stop-cock replaced. Dilute hydrochloric acid (1 : 1) is poured into the funnel. On opening the

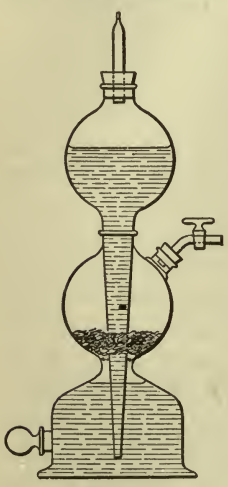

Fig. 17

stop-cock the acid will rise in the lower chamber, pass through the constriction, and come in contact with the zinc. The hydrogen generated will escape through the stop-cock. When the stop-cock is closed, the hydrogen presses downward on the acid, which is forced back into the funnel, the lowest chamber serving as a gas reservoir. In order to obtain a steady flow of hydrogen, it is only necessary to open the stop-cock.

$$Zn + H_2SO_4 = ZnSO_4 + H_2.$$

$$Zn + 2\,HCl = ZnCl_2 + H_2.$$

300 cc. flask; thistle and delivery tubes; Kipp generator; HCl (1 : 1); granulated Zn.

PROPERTIES

9. Lighter than air. — (*a*) A 1 l. beaker is suspended in an inverted position on the arm of a lecture-balance. The system is then brought into equilibrium. If a liter cylinder of hydrogen is inverted under the mouth of the beaker and the hydrogen allowed to ascend, the equilibrium of the balance will be destroyed and a deflection of the pointer be obtained. Instead of allowing the hydrogen to rise from the cylinder, it may be passed through a glass tube, the mouth of which will reach up to the bottom of the beaker. In either case, however, the balance must not be touched with the cylinder or the glass tube.

If the system be allowed to stand for a few moments, the hydrogen will rapidly diffuse out, and the balance come again to equilibrium. The removal of the hydrogen may be facilitated either by unhanging and inverting the beaker a moment, or, what is perhaps more striking, by sucking out the hydrogen through the tube used to introduce it, care being taken, of course, not to touch the beaker or the balance. If a strong suction by means of the water-pump

is maintained, the hydrogen will be rapidly withdrawn and the balance assume a state of equilibrium.

Lecture-balance ; liter beaker ; suction pump ; liter cylinder of H.

(*b*) The ascending current of hydrogen, flowing from an inverted cylinder of the gas, may be made to rotate a wheel such as is described in Ex. 36, p. 313.

A liter cylinder of the gas inverted under the buckets will cause the wheel to rotate.

Paper wheel (Fig 125, p. 313) ; liter cylinder of H.

10. Determination of the specific gravity. — By means of a balance weighing two centigrams a very satisfactory demonstration of the fact that hydrogen is approximately 14.4 times lighter than an equal volume of air may be made.

On one arm of a lecture-balance a clean, dry, graduated liter flask is suspended mouth downwards (Fig. 18) by means of a harness of fine wire. After bringing the balance into equilibrium hydrogen, dried by passing through a gas washing-bottle containing sulphuric acid, is allowed to pass through a fine tube into the inverted flask. After a few minutes the air will have been entirely replaced by the hydrogen, and it will be found necessary to place 1.14 g. on the flat bottom of the inverted flask in order to bring the system to equilibrium again.

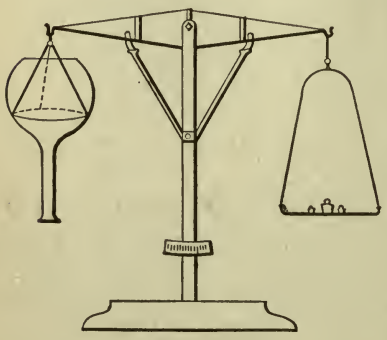

Fig. 18

Taking the weight of a liter of air at the laboratory temperature and standard barometric pressure as 1.225 g., it

will be seen that the weight of a liter of hydrogen is the difference between 1.225 and 1.14 or .085 g. On dividing the weight of air under these conditions, *i.e.*, 1.225 g. by the weight of an equal volume of hydrogen, *i.e.*, .085 g., the quotient is 14.4, or the air is 14.4 times heavier than hydrogen.

In filling the flask with hydrogen the tube should lead clear up to the bottom and should be withdrawn while the gas is still flowing through it. If the graduated flask has a long narrow neck, the rate of diffusion is so slow that there is no appreciable change of weight in bringing the balance into equilibrium. In fact, it will be some time before enough air will have diffused into the flask to disturb the equilibrium materially. If desirable, a rubber stopper can be balanced with the flask and inserted after withdrawing the tube delivering the hydrogen.

Current of dry H; liter graduated flask; lecture-balance and weights.

11. Use in balloons. — The use of hydrogen in balloons is shown by filling a small collodion balloon with the gas.

If a collodion balloon is obtainable, it is only necessary to fasten the mouth of the balloon to a glass tube through which hydrogen is passing, and after the balloon is completely filled to tie the mouth with a string. The glass tube connected with the hydrogen generator should be bent vertically upward, contain a loose 2 cm. plug of cotton wool, and be drawn down to a jet of 2 mm. diameter. The end of the jet is rounded a little in the flame to avoid cutting the thin tissue with sharp edges of glass. The collodion balloon, which should be flattened as much as possible to expel all air, is fastened well up on the shoulder of the jet so that by tightening the string and pushing the balloon down to the

point of the jet, it can be easily sealed. A balloon so filled with hydrogen rises in the air and will support a considerable length of string or thread which may be used to recover it.

H generator; collodion balloon; glass jet with cotton thread.

12. Filling a balloon under pressure. — (*a*) The collodion balloons used in the preceding experiment are short lived and somewhat expensive. The small thin rubber bags so often used on toy whistles or in games[1] are admirably adapted for experimenting with hydrogen, if provision is made to fill them with the gas under pressure, since it is necessary to fill not only the small volume of the rubber bag, but to cause its distension and thereby enclose a considerable quantity of gas. The apparatus for conducting the hydrogen into the rubber bag consists of a 1 or 2 l. bottle having a two-holed rubber stopper carrying an elbow and a jet such as is described in the preceding experiment (Fig. 19). The bottle is first filled with hydrogen either over water or by displacement, and the stopper inserted, care being taken to allow no hydrogen to escape. A rubber tube connects the water tap with the glass elbow. The rubber bag is securely fastened on the shoulder of the jet as described in the preceding experiment. By allowing the water to flow into the bottle, the gas is forced under considerable pressure out through the jet into the rubber bag, which may be distended and form a balloon some 15 cm. in diameter.

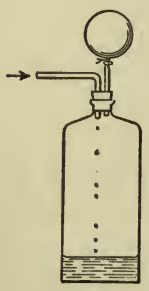

Fig. 19

H generator; 2 l. bottle; 2-holed stopper; elbow; jet; thin rubber bag.

[1] Rubber bags of this form may be obtained at any toy store.

(*b*) If the hydrogen generator is so constructed as to permit of considerable pressure, the gas may be drawn directly from the generator into the rubber balloon. A thick-walled flask is provided with a two-holed cork carrying a small long-stemmed dropping-funnel and one arm of a three-way stop-cock (Fig. 20). The zinc is placed in the flask and dilute hydrochloric acid (1 : 1) allowed to flow through the dropping-funnel into the flask. The three-way stop-cock should be so arranged that the gas escapes through the open arm of the T. The rubber balloon is tied to a jet (such as is described in the preceding experiments) which is connected by means of a short piece of rubber tubing to the stem of the T-tube. When all the air has been driven out of the generator, the three-way stop-cock should be turned so as to deliver hydrogen through the stem of the T into the balloon. It is necessary to close the stop-cock on the dropping-funnel immediately, as otherwise the gas will push up through the column of liquid and out through the funnel. When the rubber has been distended to a sufficient size, the three-way cock may be so turned as to seal the stem of the T-tube and open both arms, allowing a vent for the compressed gas inside the generator. The balloon may then be tied and drawn off the jet as described in the preceding experiment. If proper care is used in opening and closing the stop-cock, this method of filling a rubber balloon leaves very little to be desired.

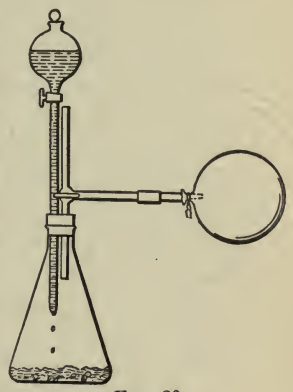

FIG. 20

Stout-walled flask ; dropping-funnel ; three-way cock ; rubber balloon.

13. Soap-bubbles blown with hydrogen. — If soap-bubbles are filled with hydrogen, they will rise rapidly and, if touched with a lighted taper, burn.

Hydrogen from a Kipp generator is first passed through a U-tube containing soda-lime slightly moistened, *i.e.*, not dry enough to have any dust, and then through a rubber tube connected with an ordinary thistle-tube. The soda-lime tube completely removes any hydrochloric acid vapor, which is quite destructive to the soap film. After dipping the mouth of the thistle-tube in the soap solution[1] and shaking off the excess of water, the bubble is started with the mouth of the thistle-tube downwards. When the bubble has attained the size of the thistle, the mouth may be pointed upwards and the hydrogen admitted as fast as desired. By means of the glass stop-cock in the Kipp generator the supply of hydrogen may be easily regulated. On giving the tube a little side shake, the bubble is released and immediately ascends. A small taper or candle fastened on the end of a long stick may be used to ignite the bubble, and it is better to hold the flame above the bubble, bringing it down so as to meet it, rather than to try to follow the bubbles and catch them as they ascend.

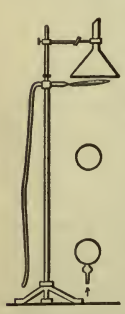

FIG. 21

A glass or preferably tin funnel 15 cm. across the top (Fig. 21) may be clamped mouth downwards to the gas pipe or other fixture over the lecture-table, and a small gas-jet burning at the end of a glass tip may be so arranged that it will burn horizontally across the mouth of the funnel. If the hydrogen bubbles are allowed

[1] *Preparation of soap solution.* — The presence of glycerine in soap solution used in blowing soap-bubbles materially increases the durability of the film.

One hundred grams of white castile (or any other pure soap) are

to rise beneath this funnel, they will ascend and come in contact with the gas flame, insuring ignition. The mouth of the funnel should be about 1.5 m. from the point of liberation of the bubble.

H generator (Kipp) ; candle at the end of a long stick ; thistle-tube ; soap solution ; soda-lime ; U-tube.

14. Conductivity for heat. — Hydrogen, in contrast to the other gases, is a good conductor of heat, and this property, which it shares in common with the metals, is well shown by lowering a jar of hydrogen over a piece of platinum wire which has been heated by means of the electric current.

A 20 mm. piece of fine platinum wire is connected with two upright copper wires fastened to a small block of wood. The copper wires should be 30 cm. long and so arranged that about a 20 cm. length stands upright from the board. They are fastened about 25 mm. apart, thus permitting the lowering of the cylinder of hydrogen over them (Fig. 22). The copper wires are connected with a battery of sufficient strength to just raise the platinum wire to incandescence. On lowering the jar of hydrogen over the glowing wire the gas will be ignited at the mouth of the jar, and the glow will nearly if not entirely disappear from the wire. On removing the jar, the glow will again appear.

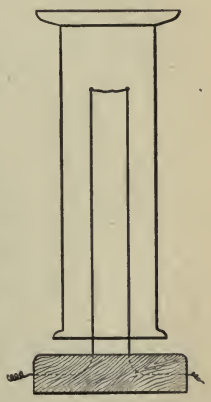

Fig. 22

cut in thin shavings and placed in a bottle with 1 l. of water. The mixture should be well shaken until a saturated solution of soap is obtained. After allowing the liquid to stand for some time the clear supernatant solution is decanted and mixed with half its volume of glycerine.

The strength of the current may be previously determined and the jar lowered before the connections are made. On switching on the current, the wire will undergo no apparent change, but on withdrawing the jar, it will become heated and ignite the hydrogen at the mouth of the cylinder. The platinum wire should be as fine as possible (such as that used in suspending Welsbach mantles), and care should be taken not to have the current strong enough to melt it.

Fine iron wire may be used in place of platinum, though it cannot be raised to as high a degree of incandescence without danger of oxidation and combustion in air. In this case it is better to lower the jar and then close the circuit and allow the iron wire to become heated on removing the jar containing hydrogen.

Fine platinum or iron wire; stout copper wire; battery; jar of hydrogen.

15. Non-conductivity for sound. — Hydrogen is a poor conductor of sound, and consequently when a bell is thrust into an atmosphere of hydrogen and then struck, very little if any sound is heard.

A small bell is fastened on the end of a stick or wire and then thrust up into a liter bell-jar of hydrogen. If the bell is then struck with a second piece of wire or a file, the sound is very much diminished.

FIG. 23

The effect is more striking if an electric bell (Fig. 23) is used. A switch should be placed in the circuit, which is closed after the bell is thrust into the jar. The box which usually covers the magnets of the bell should be removed, as it is possible to have an explosive mixture of hydrogen and air formed inside the box which might be ignited by the feeble spark produced at the contact.

Two 1 liter bell-jars; bell and wire; electric bell; battery and switch; H generator.

16. Diffusibility. — The great diffusibility of hydrogen through the walls of a porous cell may be shown by a number of striking experiments in which the variations in pressure caused by the hydrogen entering or leaving the cell are utilized.

A porous cell, approximately 12 cm. long and 5 cm. in diameter, is provided with a two-holed rubber stopper tightly fitted into its mouth. Through one hole a 5 cm. length of glass tubing of 6 mm. internal diameter is thrust, and through the other a short piece of small glass tubing about 3 mm. internal diameter. The smaller glass tube is plugged with a short piece of rubber tubing and a bit of glass rod. The larger tube is connected by means of a rubber tube with a long glass U-tube half filled with colored water. If a jar of hydrogen is now brought down over the porous cell, the pressure inside is increased and the level of the water in the U-tube will be disturbed, that in the arm connected with the porous cell falling, that in the other arm rising an equal distance.

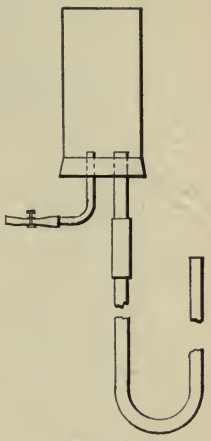

Fig. 24

Porous cup ; long glass U-tube ; ink water ; jar of H.

17. Diffusion out of a porous cup. — When the interior of the porous cup is filled with hydrogen, the diffusion outward is so rapid as to cause an internal diminution of pressure.

The jet tube is removed from the Wolff bottle in Fig. 25 and a rapid stream of hydrogen is sent into the porous cup through a small glass tube which is provided with a rubber connector and a pinch-cock. On suddenly stopping the

flow of the gas by closing the pinch-cock, the hydrogen will diffuse out of the porous cup into the air, diminishing the internal pressure inside the cup. The colored water will rise immediately in the pipette, nearly filling the bulb.

Apparatus (Fig. 25); H generator (Kipp).

18. Diffusion producing a fountain. — One neck of a small two-necked Wolff bottle is fitted with a glass tube whose lower end nearly touches the bottom of the bottle, the upper end reaching a few centimeters through the cork and being drawn down to a fine jet (Fig. 25). The Wolff bottle is then nearly filled with water colored with ink. The stem of a 100 cc. pipette is thrust through a rubber stopper in the second neck, extending nearly to the bottom of the bottle. The upper end of the pipette is connected with the porous cell described in Ex. 16. On lowering a bell-jar of hydrogen over the porous cell, the internal pressure becomes so great that the water is forced out of the Wolff bottle in a fine jet to a considerable height.

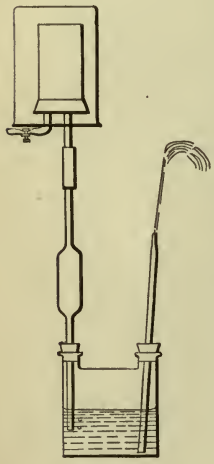

Fig. 25

Apparatus (Fig. 25); 2-necked 500 cc. Wolff bottle; 100 cc. pipette; porous cup used in Ex. 16; glass jet to fit second neck of the Wolff bottle; bell-jar of H.

19. Diffusion and its application to the fire-damp indicator. — The porous cup in Ex. 16 is connected with one limb of a U-tube which is half filled with mercury. Through the open end of the U-tube two insulated annunciator wires twisted together are thrust, the ends of which are made bare so as to give good electrical contact. One of the wires

dips some distance into the mercury, and the bare end of the other wire is held a millimeter above the mercury meniscus. On lowering the bell-jar of hydrogen over the porous cup, the internal pressure will cause the mercury in the open end of the U-tube to rise, thereby closing an electric circuit, which consists of a battery and a bell [1] or buzzer.

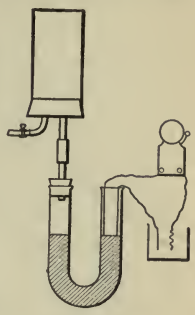

Fig. 26

The practical application of the indicator in mines may be better shown by having the bell at some distance from the lecture table; in the back part of the room, for example. A further realism may be added by covering the apparatus with a large box or closet and admitting illuminating gas into its interior, thereby creating an artificial fire-damp.

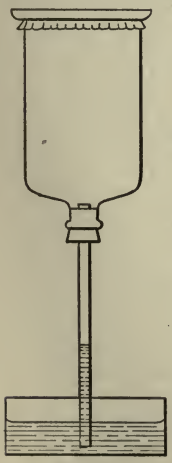

Fig. 27

Porous cup used in Ex. 16; insulated annunciator wire; battery; bell or buzzer; U-tube; Hg.

20. Diffusion through rubber. — That hydrogen also readily diffuses through a material such as rubber may be shown by means of the following apparatus (Fig. 27): —

A tubulated bell-jar is fitted with a cork and a wide glass tube 20 cm. long. A piece of thin dentist's rubber is tightly stretched over the mouth of the jar, and tightly tied on with a string passing under the rim of glass around the mouth of the jar. After clamping the apparatus in an upright position, hydrogen is conducted through a fine

[1] Dry batteries and bells or annunciators or buzzers are readily obtainable at almost any electrical supply store, at a very low price.

tube small enough to be pushed up through the larger tube into the bell-jar. When all the air is displaced, the small glass tube is withdrawn without stopping the flow of hydrogen, and a vessel of colored water is so placed that the large glass tube will dip under the surface of the water. Soon the hydrogen will begin to diffuse out through the rubber, and the colored water will rise in the glass tube.

Apparatus (Fig. 27); small tubulated bell-jar; sheet of dentist's rubber 15 cm. square; ink water.

21. Separation of hydrogen and oxygen by diffusion. — The elements of oxyhydrogen gas may be separated by diffusion by passing a slow stream of the gas through a long porous tube and testing the gas issuing at the other end after collecting it over water. The hydrogen being much more diffusible than oxygen, rapidly passes out through the walls of the porous tube, leaving the oxygen behind.

A simple apparatus for demonstrating this principle may be made by connecting the tips of two clay pipes with extra long stems by a short piece of rubber tubing. The bowl of each pipe is closed with a one-holed rubber stopper. One pipe is connected with a supply of oxyhydrogen gas, and the other with a pneumatic trough (Fig. 28). The rate at which the gaseous mixture is passed through the pipes will influence in a marked degree the composition of the gas collected at the pneumatic trough. If the rate is too

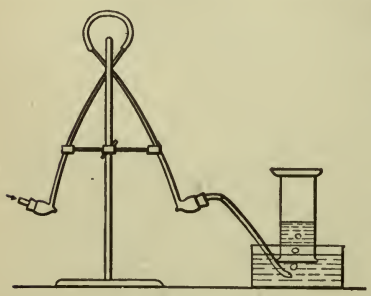

FIG. 28

rapid, the hydrogen will not have time to diffuse out, and the collected gas will still retain its explosive character. If the rate is too slow, sufficient air to dilute the oxygen will have diffused into the tube, and the collected gas will not relight a glowing splinter. A preliminary test should be made to determine the rate of bubbling in the pneumatic trough that will give the most satisfactory results. For preliminary testing not more than 30 cc. should be collected at one time. The so-called churchwarden pipes serve this purpose admirably, and they may be mounted in a double burette clamp. The oxyhydrogen gas is best held in a tubulated bell-jar fitted with a cork and a stop-cock, immersed in a pail of water. The jar is filled one-third with oxygen and two-thirds with hydrogen.

The effect of increasing and diminishing the rate of flow of the gases should be noticed after the experiment is ended. Increasing the flow causes the collected gas to explode; diminishing the flow contaminates the collected gas with air to such an extent that a glowing splinter is not rekindled.

Oxyhydrogen gas in a tubulated bell-jar; 2 churchwarden pipes; double burette clamp.

22. Combustion in air. — (*a*) The characteristic colorless hydrogen flame is best obtained by allowing gas from a Kipp generator (free from air) to burn at the tip of an ordinary blowpipe. The flame is so nearly colorless that it cannot be seen at a distance, so its presence is shown by igniting a piece of paper with it or by holding in it a fine piece of platinum wire, which will be heated to incandescence.

In case a glass jet is used, the flame will be colored yellow from the sodium in the glass. A platinum tip (Ex. 5, p. 182) may be substituted for the glass or blowpipe jet.

Blowpipe jet; fine platinum wire.

(*b*) Owing to its low specific gravity, hydrogen may be siphoned out of a glass bell-jar by means of a glass siphon tube, 8 to 10 mm. in diameter. A large bell-jar is clamped mouth downwards and the shorter limb of a glass siphon thrust up into it (Fig. 29). A rapid stream of hydrogen from a generator enters the bell-jar through the siphon tube, pushing the air down until the jar is completely filled with hydrogen. The rubber tube leading from the generator is disconnected, and the hydrogen begins to flow upwards out of the longer arm of the siphon. After the siphon is started, the gas may be lighted at the upper end, and will burn quietly until the mixture of air and hydrogen reaches the flame, when the latter will be seen to run slowly back through the siphon, igniting the explosive gaseous mixture inside the bell-jar, producing a loud though harmless report.

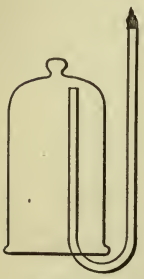

Fig. 29

Apparatus (Fig. 29); large bell-jar; siphon tube (1 cm. inter. diam.).

23. Chemical harmonica. — Hydrogen is ignited at the end of a metal blowpipe tip, which is straightened out and clamped in a vertical position, and glass tubes of varying diameters are lowered in turn some 15 cm. over the burning jet (Fig. 30). As the flame burns in the glass tubes, it produces wave motions resulting in different tones. By varying the diameters of the glass tubes and the distance that they are lowered over the burning jet, great diversity in tone may be produced. The glass tubes should not be too small in diameter. As a general rule the maximum diameter giving satisfactory results is equal to the

Fig. 30

length of the hydrogen flame; so a flame 2 cm. long would give a tone with a glass tube 2 cm. in diameter.

Straight metal blowpipe ; glass tubes, different sizes.

24. Ignition of hydrogen by platinized asbestos. — (*a*) A stream of hydrogen impinging on finely divided platinum is ignited.

A small bundle of asbestos fibre is wound in a loop at the end of a stout copper wire and moistened with a few drops of platinum chloride solution. On ignition in a Bunsen flame the platinum chloride is decomposed and the asbestos coated with a fine deposit of metallic platinum. On cooling and holding the platinized asbestos in a stream of hydrogen, it is first seen to glow, and then almost instantaneously the hydrogen is ignited. This is the principle of the self-lighting lamp of Dorbereiner.

Asbestos ; copper wire ; platinum chloride solution.

(*b*) A dry jar is filled with hydrogen by displacement and held in a slightly inclined position mouth downwards. On carefully introducing the bundle of platinized asbestos near the mouth of the jar, it will be seen that the asbestos will glow, causing a combustion of the hydrogen. If now the asbestos is carefully inserted into another dry jar of hydrogen, its introduction can be so regulated that the combustion of the hydrogen and the air will not be produced on the asbestos to such an extent as to cause the ignition of the hydrogen. The water formed by this slow combustion will be seen as white clouds at the mouth of the jar, which finally condense as moisture on the walls of the cylinder. If the bundle of asbestos is suddenly thrust up the jar into the hydrogen atmosphere, it will no longer glow, and the action ceases. On withdrawing it till it comes in contact with the zone between the hydrogen and the air, the combustion again proceeds.

Two 200 cc. dry cylinders of hydrogen ; platinized asbestos.

25. Reduction of cupric oxide by hydrogen. — A small quantity of powdered cupric oxide is placed on a porcelain crucible lid, the ring of which is pressed into a slit in a cork fastened on the end of a long iron wire. This arrangement permits of thrusting the crucible lid up into an atmosphere of hydrogen in a dry liter cylinder. The cylinder is first filled with hydrogen and clamped in a vertical position with the mouth downwards, the mouth being kept closed by holding a glass plate against it. The cupric oxide is heated from above with a small Bunsen flame till quite hot and then thrust rapidly up into the hydrogen. Soon moisture condenses on the side of the jar, and on allowing the cupric oxide to cool in the atmosphere of hydrogen for a few minutes and then withdrawing it, it will be seen to have the characteristic red color of metallic copper.

Crucible lid; cork; stout iron wire; liter cylinder of H; CuO powder.

OXYHYDROGEN GAS AND WATER

26. Formation of water by the combustion of hydrogen in air. — (*a*) By passing the products of combustion of hydrogen in air through a U-tube, a considerable quantity of water is condensed in a short time. In the apparatus (Fig. 31) one limb of the U-tube is fitted with a rubber stopper and a glass elbow connected with the suction-pump. The other limb is fitted with a cork containing the small end of a bent thistle-tube. Hydrogen, dried by passing through a U-tube containing calcium chloride, is allowed to burn from a blowpipe jet held immediately under the bulb of the thistle-tube. The rate of suction must not be such as to cause the first

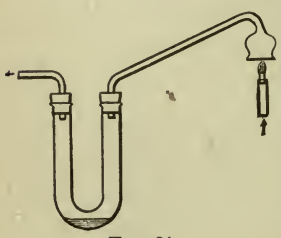

FIG. 31

limb of the U-tube to become warm enough to vaporize any water that may have condensed.

Apparatus (Fig. 31); U-tube; bent thistle-tube; metallic blowpipe jet; $CaCl_2$ drying tube.

(*b*) Dry hydrogen is allowed to escape through a blowpipe jet and a dry liter bell-jar is held over the tip to show that no moisture is deposited inside the jar. On lighting the hydrogen jet and bringing the bell-jar again over the flame, moisture will soon be deposited all over the inside of the jar. The hydrogen flame should not be high enough to generate sufficient heat to break the jar.

H generator; drying tube; blowpipe jet; liter bell-jar (dry).

27. Synthesis of water by reduction of cupric oxide by hydrogen. — A hard-glass bulb-tube may be packed full of cupric oxide, such as is used in elementary organic analysis, and weighed. After reduction and cooling in a current of hydrogen, the loss in weight may very readily be noted. The water formed may be collected in a U-tube containing glass beads or bits of pumice-stone drenched with concentrated sulphuric acid. A U-tube containing 10 g. of sulphuric acid will take up approximately 1 g. of water, though, if a U-tube having a bulb blown on one arm is used, the greater portion of the water will condense in the bulb and the sulphuric acid will absorb the uncondensed moisture. In this way several grams of water may be collected with quantitative accuracy. With careful manipulation it is not a difficult matter to make the whole experiment quantitative, *i.e.*, to determine the loss of weight of the cupric oxide and the weight of water formed. With these data a simple calculation will show that 8 parts of oxygen require 1 part of hydrogen to form water.

$$CuO + H_2 = Cu + H_2O.$$

Hard-glass bulb-tube; H_2SO_4 and pumice-stone U-tube; current of purified H; granular CuO.

28. The oxyhydrogen flame. — The intense heat of hydrogen burning in air may be still more increased by introducing a jet of burning oxygen in the centre of the hydrogen flame. The hydrogen burning under these conditions is furnished with a supply of oxygen from the air on the outside and pure oxygen for the interior, giving rise to the intensely hot oxyhydrogen flame. In burning these two gases a special form of burner is required, consisting essentially of a tube in the centre of which a fine jet is longitudinally placed. Hydrogen is admitted to the larger tube and ignited at its mouth. A gentle current of oxygen is then directed through the fine jet into the centre of the hydrogen flame. Burners specially adapted for this form of flame are much used in producing the so-called calcium or Drummond light.

In case such a burner is not obtainable the phenomenon may be studied, though less advantageously, with an ordinary gas blast-lamp. Unless a large hydrogen generator is at hand, it is more satisfactory to use illuminating gas in place of hydrogen. Oxygen may be obtained from a gasometer or from a steel cylinder. In using the lamp, illuminating gas is first admitted to the larger tube and ignited at the mouth of the burner, where it will burn with a flickering, smoky flame. Oxygen is then gradually admitted through the central air tube. As the result of the increase in temperature, the illuminating gas burns with almost dazzling brilliancy, which gradually diminishes as more oxygen is admitted. The heat is so great that only a small flame, some 5 or 6 cm. in length, can be used. Hydrogen or coal gas under considerable pressure, when used with a regular oxyhydrogen burner, will, however, give flames of any desired length. The small colorless pointed flame is extremely hot, and may be used in any of the following experiments to show its intense heat.

A bundle of iron wires or a small rat-tail file, when held in the flame, burns with great brilliancy, sending out showers of sparks. The molten globules of iron oxide fall to the table, which should be protected with a sheet of asbestos paper. The burner should be so placed that the flame is horizontal. After the iron has become well ignited, the illuminating gas may be cut off and the iron held in the jet of oxygen, where it continues to burn.

A thick copper wire is immediately melted when held in the flame.

Pieces of zinc, lead, and cadmium are readily oxidized, while silver, though not oxidized, is soon melted to a bright metallic globule, which by continued heating, may be made to boil. The silver should be placed in a small bone-ash cupel and the flame directed upon it from above. If after the silver is melted it is allowed to cool in a current of oxygen, a considerable quantity of the gas will be absorbed by the metal, and just before solidification the gas will be given off suddenly, causing a spurting out of the metal.

A piece of thick platinum wire held in the flame is soon melted to a globule, which may be shaken from the wire and allowed to drop into a dish of water placed beneath the flame.

A glass rod held in the flame is immediately melted, and drops of molten glass are allowed to fall into a cylinder of water.

Perhaps the most remarkable phenomenon that can be produced with the oxyhydrogen flame is the so-called calcium light. A piece of quicklime held in the flame is immediately raised to a brilliant incandescence of almost blinding intensity. Other highly refractory substances, such as magnesium oxide, may also be raised to incandescence when held in the flame.

In all experiments with the oxyhydrogen flame it is advisable to protect the hands with gloves and the eyes with colored glasses.

Oxyhydrogen blowpipe or burner; gas blast-lamp; cupel; O and H supply; bundle of iron wires; Cu and Pt wires; pieces of Ag, Pb, Cd; quicklime.

29. Explosion of hydrogen and air. — (*a*) A round-bottomed thick-walled "ginger-ale" bottle is marked in sevenths. Two-sevenths of its volume are filled with hydrogen over the pneumatic trough, and by lifting the mouth of the bottle and allowing the water to run out, the remaining five-sevenths are filled with air. On closing the mouth of the bottle with the thumb, and inverting it once or twice, the gases will rapidly mix, and when a taper is applied to the mouth, a sharp explosion will result. A good stout bottle should stand the force of the explosion, though, unless previously tested, it is well to cover the bottle with a towel.

Round-bottomed ginger-ale bottle; H generator.

(*b*) A bulb-tube is filled with hydrogen by allowing the gas to flow through the upper end of the tube, when held vertically, and thereby to push out the air at the bottom. When full, the rubber tube through which the hydrogen flows is removed from the upper end of the bulb-tube and a flame applied. The hydrogen, being lighter than air, rises rapidly in the tube and burns at the top. In a few minutes the air entering the bottom of the tube will have mixed with the hydrogen in explosive proportions, and the flame will strike downwards from the mouth of the tube, producing a sharp, though harmless, explosion. The internal diameter of the arms of the bulb-tube should not be greater than 5 or 6 mm. In case the tubing is larger than this, it is

advisable to choke the upward flow of the gas by inserting in the upper arm of the bulb a small piece of glass tubing thrust through a small cork, or through a short piece of rubber tubing.

Bulb-tube ; H generator.

(*c*) The preceding experiment may be performed with an equal degree of safety on a much larger scale by using a tubulated liter bell-jar, the tubulature of which is fitted with a cork containing a short piece of tubing 6 mm. in diameter (Fig. 32). The bell-jar is filled with hydrogen, either by sending a slow stream of the gas in at the top through the glass tube, thus forcing out the air at the bottom, or at the pneumatic trough, in which latter case it will be necessary to plug the glass tube temporarily by a short piece of rubber tubing and a bit of glass rod. The hydrogen is lighted at the top of the glass tube, and burns regularly, as it is forced out by the air rising from the bottom. In a few minutes the explosive mixture with air is formed, and the flame strikes downwards through the glass tube, causing an explosion in the bell-jar.

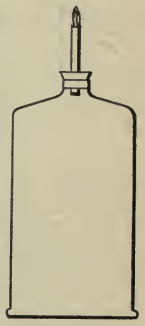

FIG. 32

Tubulated liter bell-jar ; H generator.

30. Explosion of a hydrogen generator. — (*a*) To illustrate the explosive nature of a mixture of hydrogen and air and the consequent danger of applying a lighted taper to a hydrogen generator before all the air has been driven out, a stout round-bottomed ginger-ale bottle is used for the generator, as, owing to its strength, it will suffer no damage by an internal explosion (Fig. 33). Ten grams of granulated zinc are placed in the bottle, which is clamped in an up-

right position, and 8 cc. of dilute sulphuric acid, made by adding 1 cc. of concentrated sulphuric acid to 7 cc. of water in a test-tube are poured, while still warm from the act of dilution, into the bottle. A well-fitting one-holed cork, carrying a 10 cm. length of oiled paper tube, such as is used in drinking beverages, is loosely placed in the mouth of the bottle and the tip of the paper immediately ignited. The burning paper soon ignites the explosive mixture of hydrogen and air which strikes back into the bottle, producing a sharp explosion and blowing out the cork. While care should be taken not to direct the bottle toward any one, the nature of the cork and the paper tube is such as to cause no damage.

FIG. 33

Ginger-ale bottle; 10 cm. paper tube (artificial bar straw); granulated Zn.

(*b*) A more striking illustration of the danger of explosion of a hydrogen generator by premature lighting is shown by means of the following apparatus. A 250 cc. Erlenmeyer flask, preferably of very thin glass, is fitted with a two-holed stopper carrying a thistle-tube and a glass elbow. A few scraps of zinc are placed in the bottom of the flask and 5 cc. of concentrated hydrochloric acid poured into the thistle-tube. A candle fastened on a wire is placed with its flame at the end of the glass elbow which serves as a delivery-tube for the gas. To retard the action of the hydrochloric acid on the zinc in order to afford time for lighting the candle and properly covering the apparatus,

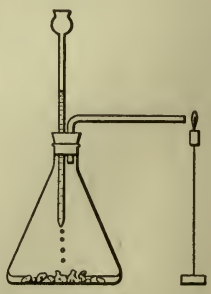

FIG. 34

the lower end of the thistle-tube is drawn out into a fine point, allowing the acid to fall drop by drop (Fig. 34). Soon the mixture of hydrogen and air assumes the right proportion for the mixture to strike back through the glass elbow into the flask, producing an explosion which will destroy the whole apparatus. In order to protect the audience, it is necessary either to cover the entire apparatus with a small wooden box, or better, to surround it with heavy glass screens. Instead of a thistle-tube, a piece of glass tubing may be used, of which the lower end is drawn out into a fine point, and the upper end, extending but a short distance above the cork, is joined with a small glass funnel by means of a rubber connector. In this arrangement the glass funnel is seldom broken.

250 cc. Erlenmeyer flask; thistle-tube; glass elbow; two-holed rubber stopper; wooden box or glass shields; candle; scrap Zn.

31. Explosion of hydrogen and oxygen. — (*a*) While the explosion of hydrogen and diluted oxygen, *i.e.*, atmospheric air, is sharp, that produced by the ignition of a gaseous mixture consisting of two parts hydrogen and one part oxygen is very violent, necessitating considerable care in its demonstration.

The round-bottomed bottle used in Ex. 29 is two-thirds filled with hydrogen over the pneumatic trough, and the remaining one-third with oxygen. On shaking the bottle for a moment, the gases become sufficiently mixed to produce a violent explosion when a taper is applied. It is advisable to wrap the bottle in two or three layers of towel. As the proportions in which the gases are mixed should be approximately correct, the bottle is roughly graduated by pasting a strip of paper on the outside indicating two-thirds of its volume.

Round-bottomed ginger-ale bottle; H and O.

(*b*) The explosion of a half liter of oxyhydrogen gas may be safely accomplished by placing a flask filled with the gas under a box, and exploding the mixture with an electric current (Fig. 35).

FIG. 35

A 500 cc. flask is two-thirds filled with hydrogen and the remaining third with oxygen. A two-holed rubber stopper carrying stout rods of brass, which extend to the centre of the flask, is then inserted in the neck. The inner ends of the brass rods should be electrically connected with a piece of very fine platinum wire (Ex. 14). The flask is then placed on a piece of asbestos paper on the table and covered with a small wooden box, which is in turn covered with a larger box. The larger box should be raised one inch from the table by small blocks of wood at each corner. Two lead wires are brought from the brass rods to the bichromate battery, which may be placed at some distance from the box. On making the connection, the platinum wire is heated to redness and the gaseous mixture is exploded. While it is not uncommon to have the small box destroyed by the force of the explosion, the large box will not be damaged. The glass flask will be reduced to a fine powder.

500 cc. flask; 2-holed rubber stopper; two brass rods; fine Pt wire; small and large wooden boxes; asbestos paper; bichromate battery; connecting wires; H supply; O supply.

32. Explosion of oxyhydrogen soap-bubbles. — (*a*) Soap-bubbles are blown with the oxyhydrogen mixture in a thistle-tube as described in Ex. 13. On being liberated they rise slowly through the air, and are easily ignited by touching with a small wax candle on the end of a stick. It is of the utmost importance that no particles of burning paper or other material should be allowed to come

in contact with the thistle-tube by which the bubbles are blown, as a dangerous explosion would result by the striking back of the explosive gaseous mixture into the holder. It is advisable to use a non-sparking material for a taper, such as a small wax candle firmly fastened on a long stick. The glass funnel used in igniting bubbles filled with hydrogen (Fig. 21, p. 52) may be used here to advantage.

Oxyhydrogen gas ; thistle-tube ; soap solution ; candle on stick.

(*b*) A small quantity of soap solution is placed in the bottom of a large iron mortar or dish, and a handful of soap-bubbles blown by directing a stream of mixed hydrogen and oxygen through it. *After removing the tube conducting the mixed gases*, the soap-bubbles are exploded by means of a candle on the end of a long stick. The concussion is so great that only a small volume of the mixed gases should be exploded at one time.

33. Electrolysis of water and the preparation of oxyhydrogen gas. — A simple apparatus for the electrolysis of water is shown in Fig. 5, p. 14, and consists of a small wide-mouthed bottle with a three-holed rubber stopper. Glass tubes carrying platinum electrodes are fitted into two of the holes, and a delivery-tube thrust into the third hole leads to a pneumatic trough. The end of the delivery-tube must not be thrust clear through the cork. The bottle is filled to overflowing with 10 per cent sulphuric acid, and the cork pushed down into the flask in such a manner that the dilute acid will fill the delivery-tube, expelling all air. Before turning on the electric current, care should be taken that the platinum electrodes are as far apart as possible in the vessel. On allowing the current from three or four bichromate cells to pass through the solution, a rapid evolution of hydrogen and oxygen takes place. The mixed gases may be collected

in a eudiometer (Fig. 11, p. 26) at the pneumatic trough. The eudiometer is transferred to another dish by placing the thumb on the bottom of the tube, and potassium pyrogallate solution is allowed to flow through the mixed gases and absorb the oxygen. When the absorption is complete, it will be found that the residual gas, which will burn quietly, is two-thirds of the original volume.

If the delivery-tube dips sufficiently beneath the surface of the water in the pneumatic trough to disengage the bubbles (to prevent the explosion from striking back into the bottle) from the delivery-tube before reaching the surface of the water, they may be exploded by holding a match or gas-jet at the surface of the water.

Electrolytic apparatus (Fig. 5, p. 14) ; eudiometer (Fig. 11, p. 26) ; four bichromate cells ; H_2SO_4 (10 per cent) ; potassium pyrogallate solution (Ex. 21, p. 26).

34. Electrolysis of water. — (*a*) The decomposition of water by the electric current with the separation of the two gases, hydrogen and oxygen, in their relative volumes two to one, is shown by the use of the apparatus (Fig. 46, p. 95) which, of the many forms of electrolytic apparatus described by Hoffmann, is the one best adapted for many experiments. The electrodes here used are made by passing small glass tubes through one-holed rubber stoppers inserted in the two lower openings of the apparatus. Through these glass tubes platinum wires are passed and fused to prevent any escape of the liquid through the glass tube. Small pieces of platinum foil are fastened to the ends of the wire, thus giving greater surface to the electrodes. The glass portion of the apparatus is mounted on a specially devised iron stand. After use the apparatus should be thoroughly rinsed with water, the rubber stoppers carrying the electrodes removed, and the glass stop-cocks either removed and tied with a

piece of twine to their respective tubes, or wedged into their openings with a piece of paper. When properly handled such an apparatus should last for years.

For the electrolysis of water the stop-cocks are closed and the open bulb filled with 10 per cent sulphuric acid, the electrodes having been tightly inserted. On opening the stop-cock the solution will rise, expelling the air, and in case it is desired to ignite the hydrogen after electrolysis, care should be taken to allow no water to enter the hole drilled through the glass stop-cock, or the bit of tube extending beyond. In case water should reach these portions, it should be removed by a strip of filter-paper rolled into a fine point, as otherwise on opening the cock drops of water will be forcibly ejected, and may easily extinguish a match or taper used to test the gas. As at best there is not a large quantity of gas to be experimented with, all precautions should be taken that none is wasted. Having filled both arms of the tube, the current from four or more cells of a bichromate battery may be sent through the apparatus, and immediately bubbles will rise from both electrodes. After a few minutes it will be observed that twice as much gas will have collected in one tube as in the other.

On holding a glowing splinter over the tube containing the smaller volume of gas, proof may be had of the presence of oxygen. The gas issuing from the other jet when opened may be ignited and shown to be hydrogen. If the tip of the glass tube is moistened with strong sodium chloride solution, the hydrogen flame will be sufficiently colored to be seen at a distance.

$$2\,H_2O = 2\,H_2 + O_2.$$

Hoffmann electrolytic apparatus (Fig. 46, p. 95); four cells bichromate battery ; 10 per cent H_2SO_4.

(*b*) The decomposition of acidulated water by the electric current may be much more simply effected by using the

apparatus shown in Fig. 36. The electrodes of this apparatus are prepared by sealing a short piece of platinum wire fastened to a piece of platinum foil into a drawn-out tube bent in the form of a hook. The tube is then filled with mercury, and the connections are made by thrusting the lead wires from a battery into the mercury in the tube. Two glass cylinders of equal diameter are filled with the acidulated water and inverted over the electrodes.

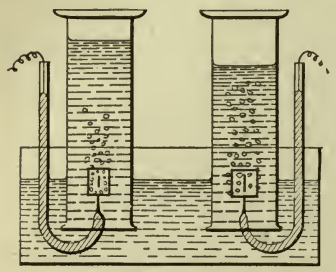

FIG. 36

The electrodes may be still further simplified by fastening pieces of platinum foil to rubber-covered copper wire. It is necessary in this case, however, to cover with paraffin all parts of exposed copper.

HYDROGEN PEROXIDE

FORMATION AND PREPARATION

35. By cooling a jet of burning hydrogen. — When a flame of burning hydrogen is suddenly cooled, small quantities of hydrogen peroxide are formed.

Hydrogen obtained from the action of zinc on dilute sulphuric acid is allowed to burn from a glass jet, 3 or 4 mm. in diameter, directed downwards upon water in a porcelain evaporating dish, which has a few pieces of ice floating in it. Two or three drops of dilute sulphuric acid should be added to the water in the dish. The jet flame is so played on the surface of the water as to cool about one-half of it, and is

FIG. 37

allowed to burn in this way for two or three minutes. The jet should be clamped in the proper position.

The liquid is tested by adding some potassium iodide-starch solution, to which a drop of ferrous sulphate solution has been added. A blue color indicates the presence of hydrogen peroxide.

H from Zn and H_2SO_4 ; ice ; KI starch solution ; $FeSO_4$ solution.

36. By the action of sodium peroxide on water. — When small quantities of water are added to sodium peroxide, oxygen is liberated, sodium hydroxide being formed (Ex. 5, p. 11). If small quantities of the peroxide are added to a large quantity of water, the oxygen is not liberated but is retained as hydrogen peroxide.

One hundred cubic centimeters of cold water, to which 5 cc. of sulphuric acid have been added, are placed in a beaker into which 2 or 3 g. of sodium peroxide are allowed to fall slowly. The liquid should be constantly stirred. After the addition of the sodium peroxide the liquid, which should still be strongly acid, will contain considerable quantities of hydrogen peroxide.

$$Na_2O_2 + 2\,H_2O = 2\,NaOH + H_2O_2.$$

37. From barium peroxide and sulphuric acid. — Concentrated sulphuric acid acts on barium peroxide to produce oxygen which under 75° C. is rich in ozone (Ex. 30, p. 31).

When, however, dilute sulphuric acid and barium peroxide are allowed to interact under conditions which maintain the mixture at a low temperature, considerable quantities of hydrogen peroxide are formed.

A handful of crushed ice is placed in a beaker and a few grams of barium peroxide stirred in with it. Enough dilute sulphuric acid is added to cover the ice, and the liquid is fil-

tered off from the insoluble barium sulphate formed. The filtrate contains hydrogen peroxide, which may be used for any of the following experiments.

With care the method with slight alterations will give a chemically pure solution of hydrogen peroxide. Such a solution is, however, readily obtained in the market.

$$BaO_2 + H_2SO_4 = H_2O_2 + BaSO_4.$$

Crushed ice; BaO_2.

PROPERTIES

38. Action on potassium dichromate. — In a dilute acidulated solution, potassium dichromate reacts with hydrogen peroxide, giving an intense blue color due to the formation of perchromic acid. This reaction serves to identify small quantities of hydrogen peroxide.

A very dilute solution of potassium dichromate, containing a few drops of sulphuric acid, is placed in a stoppered glass cylinder and 1 cc. of hydrogen peroxide is added. The liquid is colored intensely blue. If one-half of the solution is poured into a small cylinder and allowed to stand for a few moments, the color rapidly disappears.

A 1.5 cm. layer of ether is added to the remaining solution in the stoppered cylinder and the cylinder shaken. On standing, the ether will separate and will acquire the deep blue color, which will have disappeared from the liquid beneath.

100 cc. stoppered cylinder; H_2O_2; $K_2Cr_2O_7$ solution; ether.

39. Action on lead sulphide. — The oxidizing action of hydrogen peroxide in converting a sulphide to a sulphate is shown by the conversion of black lead sulphide to white lead sulphate. To a small quantity of freshly precipitated lead sulphide suspended in water a few cubic centimeters of

hydrogen peroxide are added. Immediately the black sulphide is oxidized to the white sulphate. By dipping filter-paper in a solution of lead acetate and exposing it to the fumes of hydrogen sulphide, black lead sulphide is formed on the paper. On immersing the blackened paper in hydrogen peroxide solution, the paper becomes colorless.

$$PbS + 4\,H_2O_2 = PbSO_4 + 4\,H_2O.$$

Freshly precipitated PbS suspended in water ; H_2O_2.

40. Oxidizing action on zinc and sulphuric acid. — A small piece of zinc placed in a test-tube is covered with about 10 cc. of water, and then 1 drop of concentrated sulphuric acid added. Almost immediately the evolution of hydrogen takes place, and the liquid is seen to be opaque as a result of the bubbles. On adding an equal volume of acidulated hydrogen peroxide, the gas evolution immediately ceases and the solution becomes clear.

That this cessation of the evolution of the gas may not be the result of dilution, the hydrogen peroxide should contain enough sulphuric acid to maintain the solution at or above the original percentage of acid.

$$H_2O_2 + H_2 = 2\,H_2O.$$

Acidulated H_2O_2 solution ; granulated Zn.

41. Electrolysis of hydrogen peroxide. — The rapid oxidation of nascent hydrogen by a solution of hydrogen peroxide is well shown when the electric current is used to produce an evolution of hydrogen in an acidulated solution of hydrogen peroxide.

A small cylinder is filled with a 10 per cent solution of sulphuric acid. A similar cylinder is filled with hydrogen peroxide which contains a few drops of concentrated sulphuric acid. The solutions in the cylinders are electrically con-

nected by means of a loop of platinum wire dipping into the liquid in each cylinder (Fig. 38). Electrodes consisting of small pieces of sheet platinum are dipped into each cylinder and the circuit of a bichromate battery thereby closed. If the positive electrode is immersed in the dilute sulphuric acid, a rapid evolution of hydrogen will take place and the liquid will become turbid from the ascending bubbles. The negative electrode, *i.e.*, the one immersed in the peroxide of hydrogen solution, will also give rise to a gas evolution. If now the current be reversed or the electrodes transposed, it will be found that the electrode immersed in the hydrogen peroxide solution gives no gas evolution if the battery is not too strong, while that immersed in the dilute sulphuric acid acts as before. In this case the nascent hydrogen liberated by the electrolytic action is oxidized by the hydrogen peroxide as fast as formed.

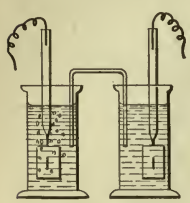

FIG. 38

When acidulated hydrogen peroxide is electrolyzed with platinum electrodes in the Hoffmann apparatus (Fig. 46, p. 95), a gas collects only in one arm of the tube, the hydrogen being oxidized as fast as formed by the hydrogen peroxide.

Bichromate battery, two or three cells; Pt wires; Pt electrodes; Hoffmann electrolytic apparatus (Fig. 46, p. 95); H_2O_2; 10 per cent H_2SO_4.

42. Bleaching action. — Hydrogen peroxide, when not too acid, bleaches organic matter readily. This bleaching action is best shown by adding to a solution of aniline red a few cubic centimeters of hydrogen peroxide. If the reagent is not too acid, the color will be discharged on boiling.

If acid is added to the hydrogen peroxide, the mixture of aniline red and the reagent can be boiled with no appreciable change in color, as the acidulated solution of the peroxide is very stable. On adding a few drops of sodium hydroxide to the hot solution, the color is instantly discharged

If the hydrogen peroxide solution is made alkaline and then added to the aniline red, the color is discharged instantly even in the cold.

Aniline red; H_2O_2.

CHLORINE

CHLORINE

PREPARATION

1. From manganese dioxide and hydrochloric acid. — Concentrated hydrochloric acid when mixed with manganese dioxide and gently heated yields chlorine.

A 2 l. flask is one-fourth filled with coarsely pulverized (not powdered) manganese dioxide. Sufficient concentrated hydrochloric acid is added to cover the solid and the mixture is heated with a low flame. A two-holed rubber stopper carrying a bulb safety-tube and a glass elbow is inserted in the neck of the flask. The gas is first conducted through a gas washing-bottle containing a small quantity of water, then through a three-way cock into a second gas washing-bottle containing sulphuric acid. The third arm of the three-way cock (Fig. 39) is connected with a flue. The bend of the safety-tube is filled with concentrated sulphuric acid or mercury. By means of the three-way cock the gas may be conducted into the flue until desired and then by turning the cock may be passed through the sulphuric acid into any apparatus.

Though no difficulty should be experienced in properly turning the three-way cock it is easy to note by the bubbling in the sulphuric acid bottle whether or no the gas is properly

directed. In case the cock should be so turned as to cut the exit of the gas off entirely, the rise of the sulphuric acid or mercury in the safety-funnel as well as the absence of bubbles in the gas washing-bottle containing water will be immediately noticed. The stopper used in the generating flask should be of rubber, and both the safety-tube and the glass elbow should fit snugly into the respective holes. Chlorine will in course of time attack rubber and render it hard and unsuited for use. By coating the under side of the stopper with collodion just before inserting it in the flask, the action of

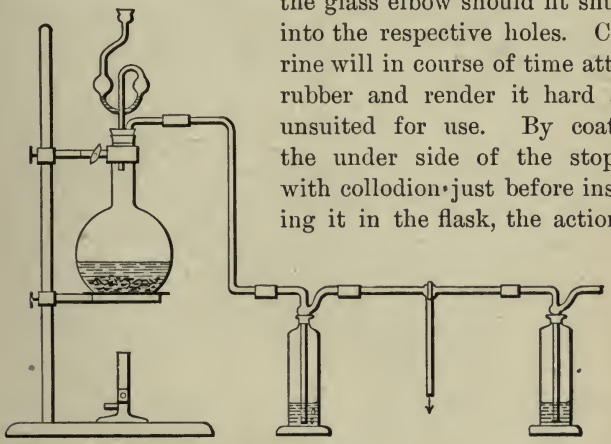

Fig. 39

the chlorine may be very much lessened. The water in the gas washing-bottle removes traces of hydrochloric acid carried over, and the gas issuing from the sulphuric acid bottle is, accordingly, quite pure. In case a flue is not at hand, the waste gas issuing from the stem of the three-way cock should be conducted through a glass tube into potassium or sodium hydroxide solution.

$$MnO_2 + 4\ HCl = MnCl_2 + 2\ H_2O + Cl_2.$$

Apparatus (Fig. 39); 2 l. flask; two gas washing-bottles; three-way cock; safety-tube; 2-holed rubber stopper; coarsely pulverized MnO_2.

2. By the action of sulphuric acid on a mixture of sodium chloride and manganese dioxide. — Instead of acting directly on manganese dioxide with hydrochloric acid, the acid may be prepared in the presence of manganese dioxide, where it is immediately oxidized with the liberation of chlorine. The apparatus used is shown in Fig. 39.

One hundred grams of finely pulverized manganese dioxide are mixed with an equal weight of sodium chloride, and the mixture is placed in the generating flask. One hundred and ten cubic centimeters of concentrated sulphuric acid are poured slowly into 100 cc. of water in a beaker and the mixture cooled. The dilute acid is then poured into the generating flask upon the manganese dioxide and sodium chloride. The flask is well shaken for a moment and the cork immediately inserted. A rapid stream of chlorine, which may be maintained by the application of a gentle heat, is obtained.

$$4\,NaCl + MnO_2 + 3\,H_2SO_4 = 2\,NaHSO_4 + Na_2SO_4 + MnCl_2 + 2\,H_2O + Cl_2.$$

Apparatus (Fig. 39) ; finely pulverized MnO_2 ; NaCl ; acid mixture (110 cc. H_2SO_4 + 100 cc. H_2O).

3. From hydrochloric acid and potassium dichromate. — Potassium dichromate oxidizes hydrochloric acid, yielding chlorine free from carbon dioxide. The apparatus (Fig. 39) is used, and 500 cc. of concentrated hydrochloric acid are poured upon 100 g. of coarsely pulverized potassium dichromate in the generating flask. By gently heating over a wire gauze a rapid stream of pure chlorine is obtained.

$$K_2Cr_2O_7 + 14\,HCl = 2\,KCl + 2\,CrCl_3 + 7\,H_2O + 3\,Cl_2.$$

Apparatus (Fig. 39) ; $K_2Cr_2O_7$.

4. By the action of a mixture of gaseous hydrochloric acid and air on heated copper salts (Deacon's process). — An in-

teresting process for the technical manufacture of chlorine is the so-called "Deacon's process," in which a mixture of air and hydrochloric acid is conducted over a heated copper salt.

Small fragments of pumice-stone, sufficient to fill a bulb-tube, are heated in an evaporating dish with a strong solution of copper sulphate or copper chloride until all water of crystallization is driven from the salt. The bulb is then filled with the prepared pumice-stone. A mixture of air and hydrochloric acid gas is obtained by allowing the gases to enter a three-necked Wolff bottle (Fig. 69, p. 159) and bubble through concentrated sulphuric acid. The mixed gases issue through the third neck of the Wolff bottle, through a glass elbow which is connected with one end of the bulb-tube. The other end of the tube is fitted with a rubber stopper and an elbow dipping into a beaker containing a solution of potassium iodide and starch. The gaseous mixture is passed through the system and no color is obtained in the beaker. On heating the bulb with a Bunsen lamp the air and the hydrochloric acid gas react in the presence of the copper salt to form free chlorine, and a blue color immediately appears in the beaker. It is necessary that the hydrochloric acid gas used in this experiment should be perfectly free from chlorine.

Instead of preparing gaseous hydrochloric acid the experiment can be more simply carried out by conducting a gentle current of air through a gas washing-bottle containing concentrated, chemically pure hydrochloric acid. In passing through the bottle sufficient hydrochloric acid gas will be taken up by the air current to react when passed through the heated bulb and will give a blue coloration in the beaker.

$$2\,HCl + O_2 = H_2O + Cl_2\,[?].$$

3-necked Wolff bottle ; bulb-tube containing pumice-stone and $CuSO_4$; HCl gas supply ; air-blast ; KI-starch solution.

PROPERTIES

5. Chlorine water. — (*a*) Chlorine is readily absorbed by water, imparting to it a greenish color and many of the properties of the gas.

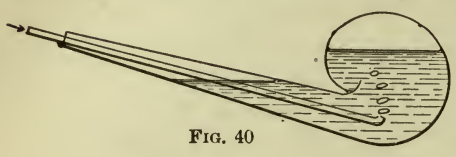

FIG. 40

A retort is completely filled with water and inverted as in Fig. 40. Chlorine is conducted through a long delivery-tube in the neck of the retort, and the body is about half filled with the gas. The absorption is quite rapid, and in a short time the water has a decided greenish color.

500 cc. retort ; Cl supply.

(*b*) The preparation of any considerable quantity of chlorine water is best effected by conducting the gas through a series of Wolff bottles (Fig. 85, p. 196) half filled with cold water. The tube through which the gas enters each Wolff bottle should dip only a little below the surface of the water. Provision should be made for absorbing any gas escaping from the system in sodium hydroxide or for conducting it to a flue.

Apparatus (Fig. 85, p 196) ; Cl supply.

6. The crystalline hydrate of chlorine. — One molecule of chlorine combines with ten molecules of water to form a white crystalline hydrate ($Cl_2 + 10\,H_2O$) which decomposes a little above zero.

A current of chlorine is conducted into 25 cc. of water in a 100 cc. flask containing a few small pieces of ice. Soon the hydrate is formed, and the liquid in the flask solidifies to a crystalline mass.

Cl supply ; ice.

7. Combustion of hydrogen in chlorine.—A jet of burning hydrogen when lowered into an atmosphere of chlorine continues to burn with a blue flame, forming hydrochloric acid.

Hydrogen is conducted through the recurved jet, Fig. 41, and lowered into a liter cylinder of chlorine. White fumes of hydrochloric acid gas rise from the mouth of the cylinder, and a piece of blue litmus paper is reddened when held in the gas.

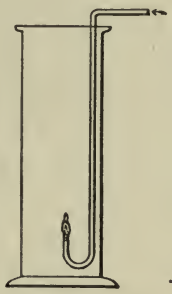

FIG. 41

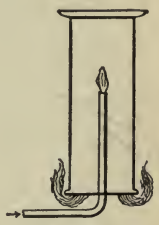

FIG. 42

A jet of chlorine burns when thrust up into an inverted cylinder of hydrogen (Fig. 42).

$$H_2 + Cl_2 = 2\ HCl.$$

Recurved jet Fig. 41; H supply; liter cylinder of Cl; litmus paper (blue).

8. Explosion of a mixture of chlorine and hydrogen.—A mixture of equal volumes of chlorine and hydrogen when ignited explodes with considerable force.

A 100 cc. stout-walled cylinder is filled with chlorine and placed mouth to mouth with a cylinder of the same size filled with hydrogen. The gases are mixed by shaking, and the cylinders are then separated and covered with glass plates. On removing the plates and applying a flame the mixture explodes with a loud report. The greatest care should be taken to keep the mixed gases away from all direct sunlight, as under the influence of bright light the gases combine with explosion.

100 cc. cylinder of Cl; 100 cc. cylinder of H.

9. Combustion of a candle. — The gaseous hydrocarbons volatilized by a burning candle will burn in chlorine with the liberation of carbon in the form of soot. A burning candle when lowered into chlorine, which should be free from any great amount of carbon dioxide, burns with a lurid, smoky flame, large quantities of soot being deposited. The flame is easily extinguished and at best burns only a few moments.

A burning splinter thrust into chlorine is immediately extinguished.

Candle on wire ; jar of Cl.

10. Action on turpentine. — When slightly warmed turpentine is introduced into chlorine, the reaction is so great that the turpentine is ignited.

Three or four cubic centimeters of turpentine are warmed in a test-tube and poured on a piece of filter-paper or cheese-cloth fastened on a wire. The cloth is then quickly lowered into a liter cylinder of chlorine. In a few moments the turpentine will become ignited and dense clouds of soot will pour out of the cylinder. Obviously the experiment should be conducted only in a good draft.

Liter cylinder of Cl ; turpentine ; filter-paper on wire.

11. Bleaching. — (*a*) Chlorine, combining, as it does, with the hydrogen of water and liberating nascent oxygen, is a powerful bleaching agent.

A piece of cloth dyed with turkey red is dampened and lowered into a cylinder of chlorine. In a few moments the color will have entirely disappeared.

The importance of having water in the operation of bleaching with chlorine may be shown as follows: A piece of turkey-red cloth which has been dried for half an hour in an air-bath immediately before use is lowered into

a cylinder of chlorine which has been especially dried by shaking the gas with 25 cc. of concentrated sulphuric acid. Care should be taken in drying the gas to have the cylinder closed with a glass plate to avoid spattering the acid. If the wire on which the piece of cloth is suspended is fastened to a piece of cardboard large enough to cover the mouth of the cylinder, the cloth may be left for some time in the gas with no appreciable change of color. If the cloth is then removed and held in steam for a few moments and again introduced into the gas, the color is almost immediately discharged.

Two cylinders of Cl (one dried by shaking with con. H_2SO_4) ; pieces of turkey-red cloth ; boiling water in a beaker.

(*b*) Chlorine bleaches many organic dyes used in preparing writing-inks, though it is without action on carbon in the graphite of the lead pencil and the lamp-black of printing-ink.

A piece of white paper, upon which several characters in printing-ink have been stamped, is marked with a lead pencil, several kinds and colors of writing-ink, and with a blue pencil. The paper is then moistened by dipping into water and thrust into a cylinder of chlorine. The printing-ink and lead-pencil marks will be unaffected by the chlorine. The blue pencil is ordinarily turned green, and the writing-inks will for the most part be entirely bleached.

Large cylinder of Cl ; piece of paper ; colored inks ; blue and black lead pencils.

12. Decomposition of chlorine water by sunlight. — In the presence of sunlight chlorine combines with the hydrogen of water, forming hydrochloric acid and liberating oxygen.

A eudiometer tube or plain glass tube sealed at one end is completely filled with strong chlorine water, inverted in a crystallizing dish containing the same liquid, and clamped

in a vertical position. If the apparatus is placed in direct sunlight, the action will soon begin, and fine bubbles of gas will collect in the top of the tube. The reaction requires some time for completion, though on standing twenty-four hours a sufficient quantity of gas will have collected in the top of the tube to be tested. A glowing splinter thrust into the gas will be rekindled. See also apparatus (Fig. 4, p. 13).

$$2\,H_2O + 2\,Cl_2 = 4\,HCl + O_2$$

Eudiometer tube ; strong Cl water.

13. Action on brass. — (*a*) Thin brass or so-called Dutch metal leaf burns when thrust into a jar of chlorine. A small piece of the leaf is fastened to a stout iron wire and lowered into a 250 cc. cylinder of chlorine. On entering the gas the leaf takes fire and burns brilliantly.

Dutch metal leaf ; jar of Cl.

(*b*) A few leaves of Dutch metal are placed in a dry, stout-walled filter-flask, which is fitted with a one-holed rubber stopper carrying a short piece of rubber tubing and a pinch-cock. In case the filter-flask has a side tube it should be securely plugged. The flask is then attached to a filter-pump, and a good vacuum is obtained. A glass tube is fastened to the free end of the rubber tube and lowered into a 500 cc. jar of chlorine (Fig 43). On opening the pinch-cock the chlorine rushes up the tube into the flask, and the Dutch metal is immediately ignited.

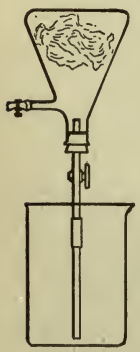

Fig. 43

A straight glass stop-cock may be advantageously used in place of the rubber tube and pinch-cock.

Filter-flask ; rubber stopper (1-holed) ; glass tube ; rubber tube and pinch-cock ; Dutch metal leaves ; jar of Cl.

HYDROCHLORIC ACID

PREPARATION

14. From the union of hydrogen and chlorine. — See Exs. 7 and 8.

15. By the combustion of chlorine in hydrogen. — The combustion of chlorine in an atmosphere of hydrogen, forming hydrochloric acid gas, may also be shown by means of the apparatus (Fig. 44).

Hydrogen from a Kipp generator is conducted through the cork in the top of the lamp chimney and ignited at the bottom. A regular stream of chlorine, which should not be allowed to bubble through a liquid, is conducted through the glass tube which extends to the centre of the cylinder. The tube is inserted in a two-holed rubber stopper which fits the base of the chimney. As the tube passes through the hydrogen flame the chlorine takes fire and may be seen to be burning at the end of the tube in the atmosphere of hydrogen. The cork is then inserted in the base of the chimney. The hydrochloric acid gas together with the excess of hydrogen issues through the open tube (15 cm. long) in the second hole of the cork. As the hydrochloric acid gas comes in contact with the air it fumes strongly. By dipping the open end of the tube a few millimeters beneath the surface of water in a beaker the hydrochloric acid will be absorbed and the excess of hydrogen will bubble through the liquid. By carefully regulating the flow of hydrogen the minimum quantity may be caused to escape uncombined.

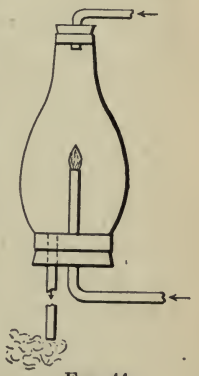

FIG. 44

It is of the utmost importance in carrying out this experiment to avoid the formation of an explosive mixture of hydrogen and chlorine inside the lamp chimney, and consequently, if the flame of chlorine should be accidently extinguished, the current of chlorine should be stopped and the cork removed, and, after filling the lamp chimney again with hydrogen, the experiment repeated.

Apparatus (Fig. 44); Cl supply; H generator (Kipp).

16. Explosion of a mixture of hydrogen and chlorine by burning magnesium. — The explosion of a mixture of equal volumes of hydrogen and chlorine may be effected by the actinic rays of a magnesium light.

A 100 cc. cylinder is filled with chlorine and placed mouth to mouth with a cylinder of equal size filled with hydrogen. The mixture of the two gases must be made by diffused daylight only or, better, by gaslight. The jars are well shaken, separated by cardboard disks, and one of them placed in front of a glass screen. The other should be covered with a towel or box to cut out the light. A strong magnesium flame is then produced in front of the screen, either by allowing magnesium powder to fall through the flame of a Bunsen burner, or by igniting one of the flash-light cartridges (Ex. 5, p. 375). As a result of the brilliant flash the mixture of gases in the cylinder on the other side of the screen explodes.

It is important that the gaseous mixture should be approximately correct as regards volume, and consequently the chlorine should not be contaminated by too great a proportion of carbon dioxide, which is likely to result from impure manganese dioxide. The flash should be very strong and a considerable quantity of magnesium must be used. The eyes of the operator should be protected by colored glasses.

An interesting addition to the experiment may be made by interposing between the light and the gases red and blue screens. When the red screen is interposed, no explosion occurs, as the actinic rays are cut off by the red glass. If the blue screen, which permits the passage of the actinic rays, is used, the explosion is produced.

$$H_2 + Cl_2 = 2\,HCl.$$

Glass screen; colored spectacles; Mg powder or flash-light cartridge; 100 cc. cylinder of H; 100 cc. cylinder of Cl.

17. By the action of sulphuric acid on sodium chloride. — Sulphuric acid reacts with sodium chloride, yielding hydrochloric acid gas.

A constant current of gas may be obtained by heating 120 g. of sodium chloride with a mixture of 100 cc. of concentrated sulphuric acid and 60 cc. of water. The diluted acid must be cooled before being introduced in the generating flask. The apparatus shown in Fig. 39, p. 81, is well adapted for this purpose. The safety-tube should be half filled with concentrated sulphuric acid and the first gas washing-bottle with concentrated hydrochloric acid. By means of a three-way cock the gas can be directed at will either into the flue or through the sulphuric acid in the second wash-bottle into a piece of apparatus. By gently heating the flask a regular current of hydrochloric acid gas, which is regulated by increasing or diminishing the size of the flame, may be obtained. The gas may be collected over mercury or, owing to its great specific gravity, by displacement.

$$NaCl + H_2SO_4 = NaHSO_4 + HCl.$$

Apparatus (Fig. 39, p. 81); NaCl; 100 cc. H_2SO_4 + 60 cc. H_2O mixed and cooled.

18. By the action of concentrated sulphuric acid on ammonium chloride. — Concentrated sulphuric acid is allowed to drop from the funnel of the apparatus (Fig. 45) upon small lumps of sublimed ammonium chloride in the flask. The issuing gas is conducted through a glass elbow in the second hole of the cork.

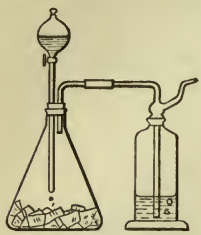

FIG. 45

This method is readily adapted for furnishing a large quantity of hydrochloric acid for a considerable period of time. In that case it is desirable to place the lumps of ammonium chloride in the middle chamber of a Kipp generator. The acid reservoir is filled with concentrated sulphuric acid.

$$2\,NH_4Cl + H_2SO_4 = (NH_4)_2SO_4 + 2\,HCl.$$

Apparatus (Fig. 45) ; Kipp generator (Fig. 17, p. 46) ; lumps of sublimed NH_4Cl.

19. By heating commercial concentrated hydrochloric acid. — Commercial concentrated hydrochloric acid consists of an aqueous solution of the gas. The gas may be expelled from the solution by gentle heat and, after being dried, used for any of the experiments described beyond. The apparatus (Fig. 39, p. 81) is suitable for this experiment when a constant stream of the gas is desired. Two hundred cubic centimeters of the strongest acid are placed in the generating flask, which is then gently heated. A rapid evolution of gas is obtained.

The gas may be made in smaller quantities by heating the aqueous solution in a flask fitted with a thistle-tube and a delivery-tube.

Apparatus (Fig. 39, p. 81) ; flask ; thistle-tube ; delivery-tube ; con. HCl.

20. By the action of concentrated sulphuric acid on concentrated hydrochloric acid. — When concentrated sulphuric acid is allowed to fall upon concentrated hydrochloric acid, large quantities of hydrochloric acid gas are liberated.

Two hundred cubic centimeters of concentrated commercial hydrochloric acid are placed in a 500 cc. flask, fitted with a two-holed rubber stopper carrying a large dropping-funnel and a glass elbow (Fig. 45). Strong sulphuric acid is allowed to enter, drop by drop, and after a few moments hydrochloric acid gas will be given off.

500 cc. flask; gas washing-bottle; rubber stopper (2-holed); dropping-funnel; elbow.

PROPERTIES

21. Generation of heat by the absorption of the gas in water. — The absorption of hydrochloric acid gas by water is accompanied by a great evolution of heat.

The bulb of a thermometer is covered with a piece of filter-paper moistened with water. If the thermometer is thrust into a jar of the gas, the water on the filter-paper absorbs sufficient gas with generation of heat to cause the mercury to rise in the thermometer some 40 to 50 degrees.

The ether thermometer described in Ex. 73, p. 174, may here be used to advantage. The bulb, which is about one-third filled with ether, is wrapped with a wet filter-paper, and the thermometer then thrust into a cylinder of the gas. Sufficient heat is generated to boil the ether. The ether vapor issuing from the end of the tube may be ignited.

Thermometer; ether thermometer (Ex. 73, p. 174); ether; 2 cylinders of dry HCl.

22. Solubility in water. — Water dissolves 450 volumes of hydrochloric acid gas at the ordinary temperature.

A dry cylinder filled with the gas is covered with a metal plate and opened under water. As the plate is slipped to

one side, the water rushes with almost explosive violence into the cylinder, filling the interior.

The absorption of the gas in water with the production of a fountain may be shown by means of the apparatus (Fig. 84, p. 195). The large flask is filled with dry hydrochloric acid gas by downward displacement, and after the cork carrying the long glass tube and small pipette nearly filled with water has been inserted, is firmly supported in an inverted position. The long glass tube is dipped under water colored blue with litmus solution. On pinching the rubber bulb of the pipette the operation is started, and the water rushes with great force through the long tube into the flask. The litmus solution is turned to a deep red by the acid.

Apparatus (Fig. 84, p. 195) ; large flask (dry) ; rubber bulb pipette (medicine dropper or ink filler) ; HCl supply; litmus solution.

23. Preparation of aqueous hydrochloric acid. — The preparation of aqueous hydrochloric acid on a large scale may be accomplished by conducting the gas through a series of Wolff bottles (Fig. 85, p. 196), provided with safety-tubes. The arrangement of the bottles is identical with that described for preparing ammonium hydroxide, with the single exception that the tubes conducting the gas into the different bottles dip only a few millimeters beneath the surface of the water.

As the absorption proceeds, currents of the heavy aqueous hydrochloric acid may be seen to be settling in the liquid in the Wolff bottles.

Series of Wolff bottles (Fig. 85, p. 196); HCl supply.

24. Electrolysis of hydrochloric acid. — The decomposition of aqueous hydrochloric acid by the electric current into equal volumes of chlorine and hydrogen is at best an unsatisfactory experiment. While the products of the decompo-

sition of water, *i.e.*, hydrogen and oxygen, are, relatively speaking, insoluble in water, chlorine is very soluble, and it becomes necessary to continue the electrolysis until the liquid is saturated with chlorine.

The electrolytic apparatus (Fig. 46) is provided with carbon electrodes, consisting of rods of electric-light carbon, preferably of a small size, inserted in rubber stoppers. Platinum electrodes, when used for this decomposition, are attacked by the nascent chlorine.

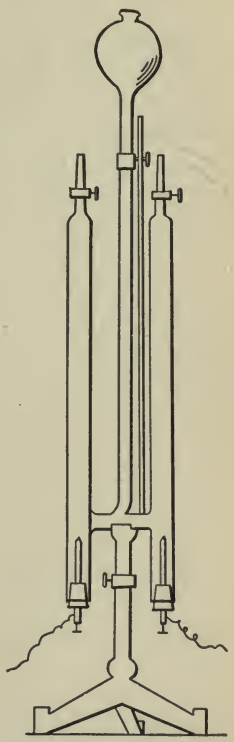

Fig. 46

The addition of small quantities of sodium chloride to the hydrochloric acid used in the experiment diminishes the capacity of the liquid for the absorption of chlorine. Concentrated hydrochloric acid is saturated with sodium chloride and the saturated liquid used in the electrolytic apparatus. Both stop-cocks being open, sufficient acid is poured into the bulb to rise in the two arms to within 2 cm. of the stop-cocks. The current from six cells of a bichromate battery is then conducted through the liquid for half an hour, leaving the stop-cocks open. While this operation as a rule should be begun before the lecture, the difference in the evolution of gases from the two poles forms an interesting experiment for showing the solubility of chlorine. At the end of half an hour the liquid in the arm of the electrolytic apparatus over the positive electrode will have

become saturated with chlorine, and by closing both stop-cocks it will be found that the two tubes fill with gas at nearly the same rate. The volume of hydrogen will probably be somewhat greater than that of chlorine, though if the stop-cock is carefully opened and the liquid allowed to rise in the hydrogen tube to the level of the liquid in the chlorine tube, after 20 or 30 cc. of gas have collected, the volumes of the gases will increase with much greater regularity. Rubber rings are advantageously placed on the arms about 15 cm. below the stop-cocks, and the actual measurement of the evolution of the gases begun when the liquids have been depressed to this level.

A piece of white paper held behind the tube containing chlorine will show the green color of the gas. On opening the stop-cock the hydrogen may be ignited as it issues from the tube, and a piece of iodo-starch paper is instantly turned blue when held in the gas issuing from the other stop-cock.

$$2\,HCl = H_2 + Cl_2.$$

Electrolytic apparatus (Fig 46); carbon electrodes; bichromate battery; HCl saturated with NaCl; KI-starch paper.

25. The electrolysis of hydrochloric acid and the collection of the mixture of hydrogen and chlorine. — The explosive mixture of hydrogen and chlorine is most satisfactorily obtained from the electrolysis of hydrochloric acid, and the mixture so formed is considerably more sensitive to the actinic rays of light than the mechanical mixture made in Ex. 16.

The hydrochloric acid is best decomposed in an electrolytic apparatus like that shown in Fig. 47. Owing to the corrosive action of nascent chlorine on platinum, it is necessary to use carbon electrodes. This may be done either by thrusting small rods of electric light carbon

through the holes of a cork, or, in case rods of small diameter are not available, pieces of carbon of the regular size may be fastened to platinum wires sealed into the glass tubes, which are filled with mercury as indicated. Inasmuch as here again the liquid being electrolyzed should be completely saturated with chlorine, the minimum quantity of acid should be used and a bottle should be selected just large enough to take the cork conveniently and not have the electrodes touch. Concentrated hydrochloric acid, saturated with salt as described in the preceding experiment, is poured into the bottle to within 2 cm. of the cork. Owing to the rise in temperature attending the electrolysis, it is advisable to place the bottle in a crystallizing dish containing cold water. The room is darkened so as to have very diffused daylight, or better only gaslight, and then the apparatus is connected with six cells of the bichromate battery. The evolution of gas begins almost immediately, though, as the major part of the chlorine is absorbed by the acid in the bottle, the first portions of the gas consist mainly of hydrogen. After the apparatus has been running for half an hour the mixed gases may be collected over salt brine. Several small cylinders and the lecture eudiometer (Fig. 11, p. 26) should be filled with the gas.

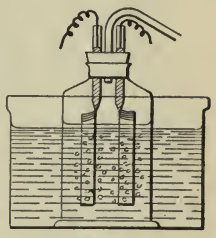

FIG. 47

At the conclusion of the experiment the current is stopped, and before exposing the apparatus to bright light, the excess of the explosive gaseous mixture remaining in the bottle above the liquid should be removed by taking out the cork.

The cylinders of the mixed gases may be exploded either by a match or by means of the magnesium light (Ex. 16).

The gas in the lecture eudiometer is reserved for use in the following experiment.

Electrolytic apparatus consisting of small wide-mouthed bottle, 3-holed cork, and carbon electrodes (Fig. 47); bichromate battery; eudiometer (Fig. 11, p. 26); salt brine; HCl saturated with NaCl.

26. Analysis of the mixture of hydrogen and chlorine obtained by the electrolysis of hydrochloric acid. — That the gas in the lecture eudiometer from the preceding experiment consists of a mixture of equal volumes of hydrogen and chlorine is shown by allowing a strong solution of potassium iodide to flow through the stop-cock into the gas. The solution immediately becomes dark-colored from the liberation of iodine, and the liquid will rise inside the tube halfway to the stop-cock. If the eudiometer is then inverted and the residual gas tested, it will be found to burn quietly, giving the hydrogen flame.

Eudiometer filled with H and Cl from the preceding experiment; KI solution.

CHLORINE MONOXIDE

27. Preparation. — When chlorine is passed over yellow mercuric oxide, the brownish oxychloride of mercury together with chlorine monoxide is formed.

Fig. 48

A slow stream of chlorine is passed through a piece of glass tubing, 50 cm. long and 8 to 10 mm. internal diameter, which is bent at right angles at a distance of 10 cm. from one end (Fig. 48). Dry mercuric oxide (the yellow amorphous modification) is placed in the tube, which is clamped in a horizontal position, the elbow dipping into a small cylin-

der. That portion of the tube containing the mercuric oxide is kept cool by allowing ice-water to drop on a piece of filter-paper laid upon it. In a few minutes the cylinder will be filled with a brownish yellow gas. Several cylinders of the same size should be filled.

A small quantity of chlorine monoxide is exploded by thrusting a burning match or hot iron wire into the mouth of a cylinder.

Sulphur flowers are sifted into a cylinder of chlorine monoxide. The reaction follows with an explosion.

A 2 or 3 mm. piece of phosphorus placed in a small deflagrating spoon is lowered into a jar of the gas. An explosion takes place.

Glass tube 50 cm. long, 8–10 mm. internal diameter; several small cylinders; ice-water; Cl generator; HgO (yellow); S flowers; P.

HYPOCHLOROUS ACID

PREPARATION

28. By the action of chlorine on mercuric oxide. — Moist, freshly precipitated mercuric oxide is covered with strong chlorine water. The oxide soon dissolves, forming a solution of hypochlorous acid.

Yellow mercuric oxide may be suspended in water in a 200 cc. flask and chlorine conducted into the liquid. The brown oxychloride is soon formed and a considerable quantity of mercuric oxide goes into solution. The clear filtrate contains hypochlorous acid.

$$HgO + H_2O + 2Cl_2 = HgCl_2 + 2HClO \quad [?].$$

200 cc. flask; Cl water or Cl generator; HgO (freshly precipitated).

29. By the action of nitric acid on calcium hypochlorite. — If a 5 per cent solution of nitric acid is carefully added to a clear solution of calcium hypochlorite, a liquid containing

calcium nitrate, calcium chloride, and hypochlorous acid is obtained which by distillation yields a colorless solution of hypochlorous acid.

Bleaching powder solution; 5 per cent HNO_3.

30. Preparation of chloride of lime. — (*a*) By conducting chlorine over moistened calcium hydroxide a mixture of calcium hypochlorite and calcium chloride, the so-called chloride of lime, is formed.

A 40 cm. length of combustion tubing is filled with calcium hydroxide which is slightly moistened. A current of chlorine, passed through a gas washing-bottle containing water, is conducted into one end of the tube and a cork

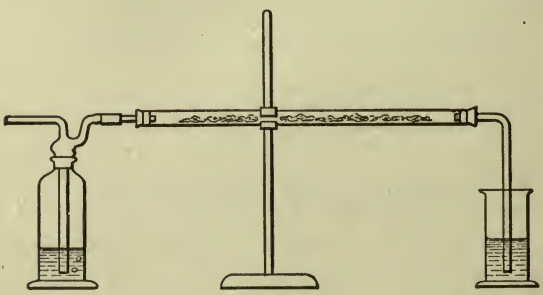

FIG. 49

carrying a glass elbow dipping into a beaker of water is thrust into the other end (Fig. 49). On account of the absorption of the chlorine by the calcium hydroxide, very little if any chlorine leaves the tube.

Apparatus (Fig. 49); 40 cm. length of combustion tubing; gas washing-bottle; Cl generator; $Ca(OH)_2$.

(*b*) A 2 l. flask is filled with chlorine and about 20 cc. of the milk of lime are introduced. On shaking the flask the chlorine is entirely absorbed, as may be noticed by the

disappearance of the green color. On the addition of an excess of concentrated hydrochloric acid, chlorine is liberated and the flask again becomes filled with the green gas.

2 l. flask ; Cl generator ; milk of lime.

31. Bleaching action of sodium hypochlorite. — Sodium hypochlorite bleaches colored cloth (turkey red). A small piece of the cloth is dipped into a solution of sodium hypochlorite in a beaker. In a few moments the color will disappear.

If hypochlorous acid is present, the bleaching is more rapid, and consequently if a small quantity of hydrochloric acid is added to the sodium hypochlorite in a second beaker, a piece of cloth dipped in the liquid is almost immediately bleached.

Sodium hypochlorite ; cloth dyed turkey red.

32. Action of hypochlorous acid on silver oxide. — Silver oxide and hypochlorous acid interact, forming silver chloride and liberating oxygen.

A few grams of the oxide are placed in a test-tube and covered with a strong solution of hypochlorous acid. On gently warming, a gas will be evolved which when tested is seen to be oxygen.

The experiment may be repeated, using cupric oxide and a solution of bleaching powder.

$$2\ HClO + Ag_2O = 2\ AgCl + H_2O + O_2.$$

HClO solution ; Ag_2O ; fresh bleaching powder ; CuO.

CHLORINE PEROXIDE

33. Preparation. — A few centigrams of finely powdered potassium chlorate are placed in a crucible which rests on the bottom of a 100 cc. cylinder. Five drops of concentrated

sulphuric acid are allowed to fall, one at a time, upon the potassium chlorate from a long glass tube bent at one end. A yellowish gas is slowly evolved and displaces the air in the cylinder. When a long iron wire is heated at one end and lowered into the gas, a sharp explosion takes place, and care should be taken to protect the face and hands from flying drops of acid (Fig. 50).

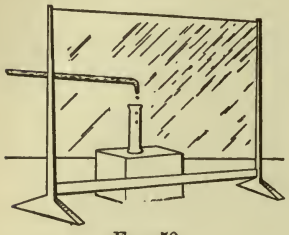

Fig. 50

In preparing chlorine peroxide the potassium chlorate may be allowed to fall into sulphuric acid. Two or three cubic centimeters of strong sulphuric acid are placed in the bottom of a small thick-walled glass cylinder and 2 g. of potassium chlorate are carefully sifted into the acid, care being taken to protect the hand with a gauntlet. The yellow gas rises in the cylinder and may be exploded with an iron wire.

Two 100 cc. stout-walled cylinders ; crucible ; bent glass tube ; glass shield ; gauntlets ; iron wire ; $KClO_3$.

34. Action of hydrochloric acid on potassium chlorate. — A concentrated solution of potassium chlorate, when heated with concentrated hydrochloric acid, turns yellow, and the gas evolved bleaches litmus paper.

If a hot glass rod is held in the gas from the test-tube, a slight explosion follows.

On the powdered salt the action of the acid is more marked, producing the mixture of chlorine and chlorine dioxide called "euchlorine."

35. Chlorine peroxide and sugar. — Equal weights of finely pulverized potassium chlorate and cane sugar are

carefully mixed on paper, avoiding all friction, and placed on an asbestos sheet or a brick in a hood. One drop of sulphuric acid is allowed to come in contact with the mixture, which is ignited and burns fiercely. The sulphuric acid may be dropped from a long bent glass tube, or the acid may be more satisfactorily added by dropping a small piece of asbestos paper in concentrated sulphuric acid and allowing the moistened paper to fall on the powdered mixture. The combustion proceeds with great rapidity, giving an intense blue potassium flame.

Asbestos sheet; $KClO_3$; sugar.

36. Combustion of phosphorus. — Phosphorus burns in chlorine peroxide, and the combustion may be made to proceed under water.

Five grams of crystallized potassium chlorate are placed on the bottom of a 100 cc. cylinder or, better, of a test-tube on foot (Fig. 51). Fifty cubic centimeters of water are then added and two or three 2 mm. pieces of phosphorus are thrown into the water.

FIG. 51

A 10 cc. pipette is half filled with concentrated sulphuric acid and the tip thrust into the cylinder until it touches the potassium chlorate crystals. As the acid comes in contact with the crystals, chlorine peroxide is evolved and the phosphorus burns.

Test-tube on foot (Fig. 51); 10 cc. pipette; crystallized $KClO_3$; 2 mm. pieces of P.

37. Explosive combustion of alcohol in chlorine peroxide. — A small quantity of chlorine peroxide is generated in a 50 cc. cylinder according to the method in Ex. 33. The cylinder is placed behind a glass screen and a few drops of alcohol are allowed to fall from a test-tube into the gaseous

mixture. A sharp explosion occurs which is likely to blow some of the acid out of the cylinder.

Glass screen; alcohol; ClO_2 in cylinder.

CHLORIC ACID

38. Preparation. — Sulphuric acid, when added to a solution of barium chlorate, forms barium sulphate and chloric acid.

Dilute sulphuric acid should be gradually added to a solution of barium chlorate until the barium is completely precipitated. On filtering off the liquid it will be found to contain chloric acid and possess strong bleaching properties.

$$Ba(ClO_3)_2 + H_2SO_4 = BaSO_4 + 2\,HClO_3.$$

Litmus; $Ba(ClO_3)_2$.

PERCHLORIC ACID

39. Preparation. — Sulphuric acid reacts with pure potassium perchlorate, liberating perchloric acid.

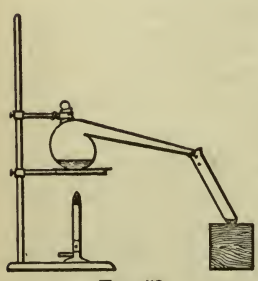

Fig. 52

Five grams of chemically pure potassium perchlorate are heated gently with 12 cc. of pure concentrated sulphuric acid in a 100 cc. tubulated retort (Fig. 52). The powder is placed in the flask and the concentrated acid poured through a funnel, the introduction of acid into the neck of the retort being thus avoided. The retort is then gently heated by a low flame. A high temperature is as undesirable as it is unnecessary, since the perchloric acid distils at about 110°. A few drops

of an oily, strongly fuming distillate are obtained in the test-tube used as a receiver.

$$2\ KClO_4 + H_2SO_4 = K_2SO_4 + 2\ HClO_4.$$

100 cc. tubulated retort; c. p. $KClO_4$; c. p. con. H_2SO_4.

40. Bleaching action. — One drop of the distillate obtained in the preceding experiment is allowed to fall into a test-tube containing 3 or 4 cc. of water. The diluted perchloric acid, when added to a solution of indigo, bleaches it immediately.

Perchloric acid; indigo solution.

41. Combustion of charcoal. — Perchloric acid is a strong oxidizing agent, and when a small piece of charcoal, heated to glowing, is dropped into a test-tube containing a few drops of the acid, the charcoal burns.

Perchloric acid; charcoal.

24. Action on organic matter. — A drop of perchloric acid on the end of a glass rod is spread on a piece of filter-paper. The acid destroys the paper completely, cutting a hole in it and igniting it. By holding the paper high above the flame and gently warming it the ignition may be immediately accomplished.

A drop of the perchloric acid is spread on the stem of a match held with pincers. The match is lighted, and when the flame reaches the part covered with acid, the combustion is much more rapid, and the flame sputters.

BROMINE

BROMINE

1. Preparation from potassium bromide. — Metallic bromides, when heated with sulphuric acid in the presence of an oxidizing agent such as manganese dioxide, give up all their bromine. This reaction, used in the technical preparation of the element, is easily carried out on a small scale.

Three and one-half grams of powdered potassium bromide are mixed with 7 g. of finely pulverized manganese dioxide and introduced into a 500 cc. retort. Fifteen cubic centimeters of concentrated sulphuric acid and 90 cc. of water are mixed and carefully introduced, care being taken to get none of the acid into the neck of the retort. After the mixture is thoroughly stirred the retort is placed on a ring-stand and its neck thrust deep into a 500 cc. flask, which can be externally cooled by immersion in water. On gently heating the contents of the retort the bromine is liberated, filling the retort with deep reddish brown fumes, which distil over and condense in the flask to a heavy, dark fluid, bromine, covered with a lighter layer of bromine water. Owing to the volatility of the bromine the flask should be cooled with ice-water. (Fig. 93, p. 223.)

$$2\,KBr + MnO_2 + 2\,H_2SO_4 = K_2SO_4 + MnSO_4 + 2\,H_2O + Br_2.$$

500 cc. retort; 500 cc. flask; ice-water; KBr; MnO_2.

2. Solidification by cold. — Bromine readily freezes in a mixture of salt and ice to a dark, crystalline mass which has a dull metallic lustre. Five cubic centimeters of bromine are placed in a thin-walled test-tube and immersed in a freezing-mixture of salt and ice. After a few minutes the bromine will have solidified. The tube is warmed a little in the hand, and the solid lump of bromine still retaining the shape of the test-tube shaken out upon a white plate.

Ice and salt; Br.

3. Vaporization. — Bromine is very volatile, giving rise to a deep reddish brown vapor. This is best shown by pouring 5 drops of liquid bromine into a dry 500 cc. flask, the mouth of which is covered with a small watch-glass. On gently warming, the vapor rises, displacing the air, and fills the flask with deep-colored fumes.

500 cc. flask; watch-glass; Br.

4. Bromine water. — Five cubic centimeters of bromine are poured into 200 cc. of water in a 250 cc. flask. The bromine, by reason of its great specific gravity, sinks to the bottom of the flask. On corking the flask and shaking, the bromine is dissolved in the water.

5. Action on arsenic and antimony. — When either antimony or arsenic is allowed to come in contact with liquid bromine, a vigorous reaction occurs, the metal burning on top of the bromine.

One cubic centimeter of liquid bromine is placed in a test-tube which is inserted in the mouth of a wide-mouthed bottle (Fig. 109, p. 262). A 2 or 3 mm. piece of antimony or arsenic is dropped into the liquid. In case the heat is sufficient to break the tube, the bromine will fall into the wide-mouthed bottle and there do no harm.

Apparatus (Fig. 109, p. 262); As; Sb; Br.

HYDROBROMIC ACID

FORMATION AND PREPARATION

6. By heating a mixture of hydrogen and bromine. — If hydrogen is allowed to bubble through bromine and is then ignited, sufficient bromine vapor will have been carried with the hydrogen to combine with it and form hydrobromic acid.

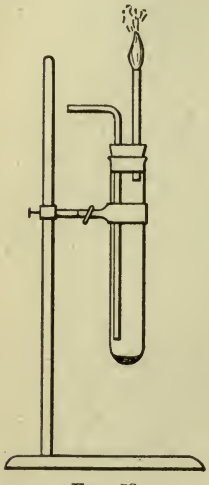

FIG. 53

A simple arrangement for this experiment consists of a large test-tube fitted with a two-holed rubber stopper. Hydrogen is allowed to pass through a glass elbow thrust through the stopper down to the bottom of the test-tube in which a few drops of bromine are placed. An open glass tube is thrust through the other hole in the cork and serves as a jet. After sufficient hydrogen has passed through the apparatus to drive out all air, the gas is lighted at the open tube. Clouds of hydrobromic acid vapor will appear, the quantity of which may be increased by gently warming the bromine in the test-tube (Fig. 53).

H generator ; Br.

7. By the action of bromine on hydrogen sulphide. — Bromine unites with the hydrogen of hydrogen sulphide, forming hydrobromic acid and setting free sulphur. By filtering off the sulphur a dilute solution of hydrobromic acid may be obtained.

Hydrogen sulphide gas is conducted into a 100 cc. Erlenmeyer flask containing 20 cc. of strong bromine water. In a

few minutes the solution will have become decolorized and large quantities of sulphur will have separated. By adding a few more drops of bromine from time to time, the reaction may be continued.

$$H_2S + Br_2 = 2\,HBr + S.$$

100 cc. flask; H_2S generator; Br water.

8. From sulphuric acid and potassium bromide. — Dilute sulphuric acid liberates hydrobromic acid from bromides in a pure form containing but small quantities of bromine vapor or sulphur dioxide. This method is very satisfactory for the preparation of gaseous hydrobromic acid.

Ten grams of potassium bromide are placed in a 300 cc. Erlenmeyer flask fitted with a dropping-funnel and a glass elbow (Fig. 54). A mixture is made of 3 volumes of concentrated sulphuric acid and 1 volume of water by pouring the acid gradually into the water and cooling the mixture. Sufficient of this mixture is added to cover the potassium bromide and the contents of the flask are very gently warmed. The gas is steadily evolved and may be collected in cylinders by displacement. The gas regulation is easily controlled by varying the heat applied to the flask. If the gas is not perfectly colorless, the small quantity of bromine vapor may be removed by conducting the gas through a U-tube filled either with red phosphorus or with glass beads or pumice-stone drenched with concentrated hydrobromic acid solution.

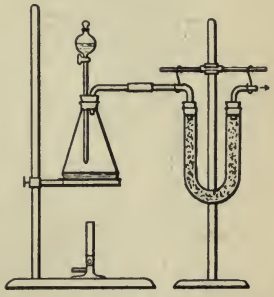

FIG. 54

$$2\,KBr + H_2SO_4 = K_2SO_4 + 2\,HBr.$$

300 cc. Erlenmeyer flask; dropping-funnel; KBr; H_2SO_4 (3 : 1); U-tube; red P; con. HBr solution.

9. From bromine and naphthaline. — Bromine reacts with naphthaline, liberating hydrobromic acid gas. This reaction is very satisfactory for preparing the gas.

Fifteen grams of naphthaline are covered with 20 cc. of kerosene in a 500 cc. distilling flask. A small dropping-funnel, the tip of which should dip beneath the liquid, is inserted in a one-holed stopper in the neck of the flask (Fig. 55). Fifteen to 20 cc. of bromine are placed in the dropping-funnel and allowed to flow very slowly into the flask. The reaction begins almost immediately, and the hydro-

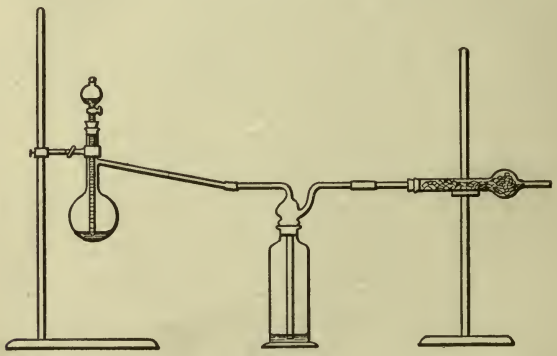

FIG. 55

bromic acid gas, which carries with it slight traces of bromine vapor, should be purified by conducting it through a gas washing-bottle containing not more than 10 cc. of a strong aqueous solution of hydrobromic acid in which a small quantity of red phosphorus is suspended. The issuing gas will be perfectly colorless. While the heat of the reaction is often sufficient to liberate the hydrobromic acid gas, the flask may advantageously be very gently warmed with a low, smoky flame. The gas washing-bottle may be replaced by a U-tube filled with red phosphorus. The puri-

fied gas is dried by conducting it through a tube containing calcium chloride.

500 cc. distilling flask; 100 cc. dropping-funnel; gas washing-bottle; $CaCl_2$ drying-tube; U-tube containing red P; naphthaline; Br; kerosene; red P; con. HBr.

PROPERTIES

10. Hygroscopic nature. — On opening a cylinder of hydrobromic acid the gas fumes in the air.

11. Action with litmus. — A piece of moistened blue litmus paper thrust into a jar of the gas will be reddened.

12. Action with ammonia. — A few drops of strong ammonium hydroxide are poured on a test-tube brush, which is then lowered into a jar of the gas. Dense white fumes of ammonium bromide are formed.

13. Solubility in water. — A jar of gaseous hydrobromic acid is opened under water. The solubility is so great that the water rushes into the cylinder with considerable violence.

14. Decomposition by chlorine. — Two cylinders of equal size are filled, one with hydrobromic acid gas, and the other with chlorine. The cylinder containing chlorine is placed mouth downwards on the top of the cylinder containing hydrobromic acid. On slipping out the glass plates between the cylinders, the gases mix and the brown vapors of bromine are set free. After standing for a few minutes, or, more rapidly by shaking, both jars become filled with bromine vapor.

If a slow stream of chlorine is passed through the recurved jet (Fig. 41, p. 85), and lowered into a jar of hydrobromic acid gas, brown fumes will immediately appear.

Jar of HBr gas; jar of Cl; Cl supply; recurved jet.

IODINE

IODINE

PREPARATION

1. From potassium iodide, manganese dioxide and sulphuric acid. — Potassium iodide when acted upon by sulphuric acid in the presence of an oxidizing agent such as manganese dioxide is completely decomposed, the iodine being liberated.

Three and one-half grams of potassium iodide are mixed with 7 g. of finely pulverized manganese dioxide, and placed in a 500 cc. tubulated retort. One hundred cubic centimeters of cold dilute sulphuric acid (made by adding 17 cc. of the concentrated acid to 85 cc. of water) are added, and the mixture thoroughly stirred. The retort is gently heated, and vapors of iodine soon appear and condense in crystals on the neck of the retort and in a flask through the neck of which the mouth of the retort is thrust. No external cooling of the flask is necessary, and care should be taken not to heat the mixture too much.

$$2\,KI + MnO_2 + 2\,H_2SO_4 = K_2SO_4 + MnSO_4 + 2\,H_2O + I_2.$$

500 cc. tubulated retort ; 500 cc. flask ; MnO_2 ; KI ; mixture of 17 cc. conc. H_2SO_4 and 85 cc. water.

PROPERTIES

2. Melting iodine. — Iodine melts at 114° to a brown liquid which solidifies at ordinary temperatures to a heavy metallic-appearing mass.

Three grams of iodine are carefully heated in a clean, dry test-tube, until completely melted. On allowing it to cool, it solidifies, and by carefully breaking the test-tube the solid mass, which has taken the form of the interior of the tube, may be placed on a plate, where its dark color and metallic appearance are readily observed.

3. Distillation. — (*a*) A few grams of iodine are heated in a small tubulated retort, the neck of which is thrust into the mouth of a large glass cylinder. On boiling the iodine, the vapors partially condense in the neck of the retort, which should be as wide as possible, and care should be taken that it does not become clogged. The vapor issuing from the neck of the retort falls by reason of its great weight into the cylinder. As the vapors descend and become cooled, the iodine solidifies in minute particles, which fall as a shower of glistening crystals to the bottom and sides of the cylinder (Fig. 56).

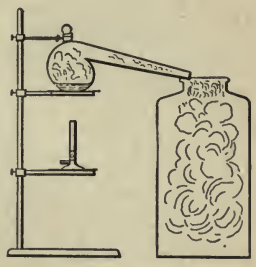

FIG. 56

250 cc. tubulated retort; 4 or 5 l. cylinder; I.

(*b*) When iodine vapor is cooled in the air, the iodine condenses in the form of minute crystals.

Two grams of iodine are heated in a test-tube to boiling, and the dense violet vapor poured out on a sheet of white paper. The iodine settles on the paper in the form of a shower of fine crystals.

4. Volatilization at ordinary temperatures. — Iodine vaporizes at ordinary temperatures, as may be seen by dropping one or two small crystals of iodine into a 3 or 4 l. flask, in the neck of which a piece of paper moistened with starch solution is suspended. After a few minutes the paper will be turned blue by the vaporized iodine.

3 or 4 l. flask ; I crystals ; starch solution.

5. Solubility in alcohol. — Alcohol dissolves iodine readily, with the formation of a deep reddish brown solution, the so-called tincture of iodine. Two and a half grams of iodine crystals are placed in the bottom of a glass cylinder and 25 cc. of alcohol poured over them. On stirring the mixture with a rod, the iodine will be entirely dissolved.

6. Solubility in carbon disulphide and chloroform. — A few crystals of iodine added to either of these reagents dissolve readily, forming a violet color, in strong contrast to the color iodine gives with ether or alcohol.

Iodine water is shaken in a stoppered cylinder with 5 cc. of carbon disulphide. The heavy solvent extracts the iodine from the iodine water, which becomes clear, and settles to the bottom of the cylinder as a violet-colored liquid.

100 cc. stoppered cylinder ; CS_2 ; $CHCl_3$; I ; I water.

7. Iodine and starch. — (*a*) Iodine forms with starch paste a deep blue color. The delicacy of this reaction is such as to cause its use in many operations in analytical chemistry.

A few drops of starch paste are added to a liter of water. Iodine water which contains but a small amount of iodine is allowed to fall drop by drop into the solution. A very few drops will produce a deep blue color, thereby showing the extreme delicacy of the reaction.

The color is discharged by an excess of chlorine water, by heat, and by alkalies.

Starch paste ; I water.

(*b*) *Effect of heat.* — A few cubic centimeters of the blue liquid are vigorously boiled in a test-tube for three or four minutes. Even before the liquid fairly boils, the color is completely discharged. On allowing the test-tube to stand it will be seen that the color does not return.

If the blue solution, containing a slight excess of iodine rather than of starch, is heated gently until the color is just discharged, and then allowed to cool, the blue color will return. This moderate heating is best carried on by immersing the test-tube containing the blue solution in a beaker of water which has just been brought to a boil, and removed from the flame. The color will shortly disappear, and if the tube is then immediately immersed in a beaker of cold water, the blue color will return.

On boiling, the iodine liberated from the starch compound by heat is volatilized with the water vapor passing off into the air. If the blue solution is heated in a distilling flask, and the vapor condensed in a long glass tube, which acts as an air condenser (Fig. 57), the liquid dropping from the end of the tube will produce a blue color in a beaker of starch paste placed beneath it. On cooling, it will be found that the residue in the distilling flask will not regain its original blue color, the iodine having been driven off by boiling.

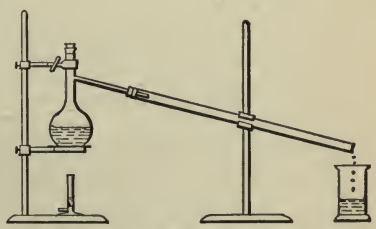

FIG. 57

250 cc. distilling flask ; long glass tube ; starch solution ; I.

8. Union with potassium. — On gentle warming, potassium and iodine unite with explosive violence.

A few crystals of iodine are placed in a dry test-tube, and a 3 mm. piece of clean, dry, metallic potassium added. When warmed, the elements unite with a slight explosion, the flame possessing the characteristic violet color of the potassium flame. Care should be taken to protect the face and hands from the results of the reaction.

$$2\ K + I_2 = 2\ KI.$$

Screens ; gauntlets ; K ; I.

9. Union with zinc dust. — Zinc dust and iodine unite in the presence of moisture with the evolution of heat.

One gram of zinc dust is intimately mixed with 5 g. of finely powdered iodine in a dry beaker. One or two drops of water are allowed to fall into the beaker, when the reaction begins, the heat generated vaporizing a large quantity of iodine. A crystalline sublimate of iodine on the walls of the beaker is obtained.

The reaction may be carried out with a similar result by using iron powder in the place of zinc dust.

$$Zn + I_2 = ZnI_2.$$

Dry beaker ; Zn dust ; powdered I.

10. Union with mercury. — Mercury and iodine unite with incandescence to form mercuric iodide.

A few globules of mercury are heated to boiling in a test-tube clamped in a vertical position. The flame is then removed and a few crystals of iodine dropped into the hot mercury. A feeble flame is observed, and the iodide of mercury sublimes in a crystalline mass on the sides of the tube.

If both elements are in the gaseous form, the union is still more vigorous. The mercury is heated in a test-tube

as before, and the lamp turned down to give a flame just sufficient to keep the mercury boiling. A few crystals of iodine are brought to a boil in another test-tube and the vapors poured upon the boiling mercury. The union is accompanied with a flame, the product being deposited as a colored crystalline sublimate.

$$Hg + I_2 = HgI_2.$$

Dry test-tubes ; Hg ; I.

11. Union with phosphorus. — Iodine and phosphorus unite, even in the cold, with sufficient energy to ignite the phosphorus.

The reaction between iodine and phosphorus may be readily shown by placing a few crystals of iodine on a brick and carefully placing a small piece of dried yellow phosphorus on top of the crystals. The phosphorus is almost immediately ignited.

Brick ; yellow P ; I crystals.

HYDRIODIC ACID

FORMATION AND PREPARATION

12. From hydrogen and iodine. — Hydrogen, mixed with iodine vapor and passed through a heated tube, unites with the iodine to form hydriodic acid.

A few crystals of iodine are placed in the bulb of an ordinary bulb-tube with long arms through which hydrogen from a Kipp generator is passed. A strip of blue litmus paper held in the issuing gas is unacted upon, showing the absence of acid fumes. The open arm of the bulb-tube is heated to a low red heat by means of a Bunsen burner, and the iodine in the bulb is then gently warmed, subliming

some of the vapors into the atmosphere of hydrogen. The issuing gas will now form white fumes of hydriodic acid and a strip of moistened blue litmus paper will be immediately reddened. A piece of paper moistened with potassium dichromate solution is instantly changed in color by the reducing action of the hydriodic acid. On igniting the gas as it issues from the bulb-tube, the heat of the burning hydrogen will be sufficient to decompose the hydriodic acid with the liberation of free iodine, which will appear as a violet vapor rising from the flame. A white paper may be held behind the flame to serve as a background for the violet vapor. If a piece of cold porcelain, such as an evaporating dish, is held in the flame, the iodine liberated will be deposited as a brownish yellow mass.

$$H_2 + I_2 = 2\,HI.$$

Bulb-tube; H generator; I; $K_2Cr_2O_7$ solution.

13. From iodide of phosphorus and water. — Water reacts with iodide of phosphorus, liberating hydriodic acid gas.

In preparing the iodide of phosphorus, 2 g. of yellow phosphorus are cut in pieces of approximately .5 g. each, carefully dried between filter-paper and dropped one at a time on top of 22 g. of iodine crystals placed in the bottom of a 100 cc. Jena glass Erlenmeyer flask. The phosphorus should be dropped as near the middle of the flask as possible, and the second and succeeding pieces should not be added until the reaction has entirely ceased. At the end of the reaction the flask will contain a liquefied mass of phosphorus iodide.

After the flask and its contents have become perfectly cold, 6 cc. of water are added, and a cork carrying a glass elbow is inserted in the neck of the flask. A purifying

U-tube containing red phosphorus should be connected with the glass elbow, the issuing gas being collected in dry cylinders (Fig. 58). The gas evolution in this operation is quite rapid, and provision should be made for filling several clean, dry jars. As the evolution of gas diminishes in rapidity, a very small flame may be used to heat the contents of the flask, which at the end of the operation will have become perfectly clear. The rapidity of the reaction after the addition of water is so great that it is advisable to wait a moment before inserting the cork in the neck of the flask.

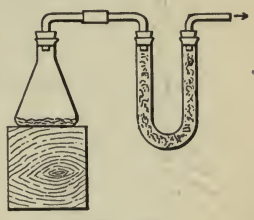

Fig. 58

100 cc. Jena glass Erlenmeyer flask; U-tube with red P; I; P (yellow).

14. By the action of iodine on rosin. — In the complex reaction obtained by heating a mixture of iodine and rosin a considerable quantity of hydriodic acid gas is formed.

Fifteen grams of finely powdered iodine are mixed with an equal bulk of finely powdered rosin. To regulate the reaction more satisfactorily, the mixture is intimately rubbed with an equal volume of pulverized quartz or very fine sand. The powder is then placed in a dry 100 cc. Jena glass Erlenmeyer flask fitted with a one-holed cork and a glass tube, 7 mm. wide, bent so as to extend nearly to the bottom of a vertically clamped test-tube (Fig. 59). The test-tube is fitted with a two-holed stopper, in the second hole of which a glass elbow is inserted. On heating the flask, hydriodic acid gas is liberated, mixed with considerable quantities of free iodine and an oily liquid, which condenses in the test-tube. To purify the issuing gas it should first be conducted

through a U-tube filled with red phosphorus, and then through a calcium chloride tube. This method gives a very

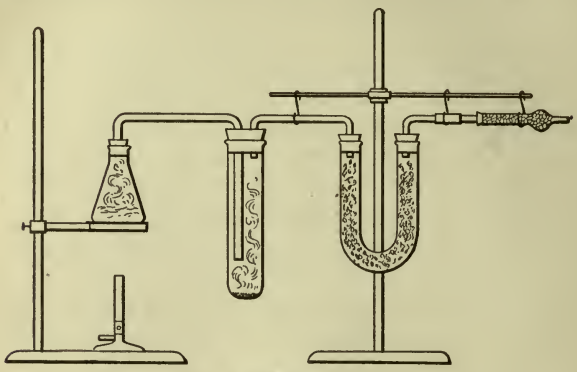

FIG. 59

good yield of hydriodic acid gas and is strongly to be recommended.

100 cc. Jena glass Erlenmeyer flask; U-tube filled with red P; $CaCl_2$ drying-tube; powdered quartz or very fine sand; powdered rosin; I.

15. By dissolving the gas in water. — A very simple method of obviating the difficulties attending the absorption of this gas in water is that of conducting the gas through the stem of an inverted funnel, the mouth of which is thrust a few millimeters under the surface of the water in a crystallizing dish. If back suction occurs, air will be drawn under the edge of the funnel before the water has risen in the cone of the funnel as far as the stem.

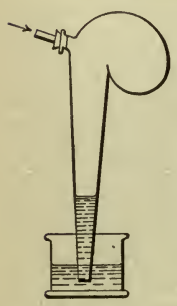

FIG. 60

The funnel may be replaced by a retort.

PROPERTIES

16. Decomposition by heat. — (*a*) The decomposition of hydriodic acid gas by heat, its great specific gravity, and the fact that it is a non-supporter of combustion, are shown by pouring 500 cc. of the gas from a cylinder upon a Bunsen flame about 2 cm. high. Iodine is set free in the form of a violet vapor and the flame is extinguished. As the hydriodic acid gas comes in contact with the air, it fumes strongly from the absorption of moisture.

500 cc. cylinder of HI.

(*b*) If a glass rod is strongly heated and then suddenly thrust into a jar of the gas, a combustion is observed and iodine vapor is liberated.

17. Acidity. — On opening a jar of hydriodic acid, dense fumes like those observed about the mouth of a concentrated hydrochloric acid bottle, are formed. If a piece of moistened blue litmus paper is held over the mouth of the jar, it will instantly be colored red.

A rod moistened with ammonium hydroxide gives heavy white fumes of ammonium iodide which partially sink into the cylinder and remain suspended in the heavy gas. A strip of paper moistened with potassium dichromate solution is instantly turned black.

Cylinder of HI gas; $K_2Cr_2O_7$ solution.

18. Solubility in water. — When a cylinder of the dry gas is opened under water, the gas is rapidly absorbed, the water rising to take its place. It is advisable to use a metal disk to cover the mouth of the cylinder, as the suction is so great as to break a glass plate.

Metal disk; cylinder of HI gas.

19. Oxidation by nitric acid. — The oxidizing action of nitric acid on gaseous hydriodic acid is so great as to produce a flame.

Three cubic centimeters of hot fuming nitric acid are poured from a test-tube into a cylinder of hydriodic acid gas. A red flame proceeds from the cylinder and large quantities of violet iodine vapor are set free.

100 cc. cylinder of HI gas ; screen ; gauntlets ; fuming HNO_3.

20. Combustion of oxygen in hydriodic acid gas. — A gentle stream of oxygen is passed through a rubber tube which dips under water to determine the rate of flow of the gas. The rubber tube is then quickly slipped over the end of a bent glass tube whose end has been heated very hot. On suddenly thrusting the tube into a jar of hydriodic acid gas, the oxygen takes fire and burns, setting free large quantities of iodine.

$$4\ HI + O_2 = 2\ H_2O + 2\ I_2.$$

O supply ; cylinder of HI gas.

21. Decomposition of gaseous hydriodic acid by means of chlorine. — (*a*) Chlorine reacts with hydriodic acid gas, forming hydrochloric acid gas and liberating iodine. In the presence of an excess of chlorine, the liberated iodine combines with the chlorine to form crystals of yellow iodine trichloride.

A current of hydriodic acid gas, prepared as in Ex. 14, is dried by being passed through a calcium chloride tube, and is then conducted through the recurved jet (Fig. 41, p. 85) into a 500 cc. cylinder of chlorine. If the end of the jet has been warmed a little, the issuing gas becomes ignited as it enters the chlorine, and burns, liberating iodine as a violet vapor. The iodine immediately combines with

the excess of chlorine, and the walls of the cylinder become covered with yellow iodine trichloride.

A current of chlorine, conducted through the recurved jet into a cylinder of hydriodic acid gas, likewise becomes ignited, liberating iodine. As chlorine continues to be introduced, the iodine combines with it, and is deposited on the walls of the cylinder in yellow crystals of iodine trichloride.

$$2\ HI + Cl_2 = 2\ HCl + I_2.$$

$$I_2 + 3\ Cl_2 = 2\ ICl_3.$$

HI supply ; $CaCl_2$ tube ; recurved jet (Fig. 41, p. 85) ; 500 cc. cylinder of Cl ; Cl generator ; 500 cc. cylinder of HI.

(*b*) Two glass cylinders are filled, the one with chlorine, the other with hydriodic acid gas, and the cylinder containing chlorine is placed mouth downwards on top of the cylinder containing hydriodic acid gas. Glass plates separate the two cylinders. On slipping out the glass plates, and allowing the mouths of the cylinders to come together, the chlorine and hydriodic acid gas interact, iodine being liberated. A slight flame is noticed at first, though there is not enough heat generated to force the gases out into the room. As the hydriodic acid gas is the heavier, the progress of its diffusion into the lighter chlorine may be noted by the color change of the deposition on the walls of the chlorine cylinder. At first in the upper cylinder, where there is an excess of chlorine, the deposition consists chiefly of iodine trichloride, a yellow crystalline compound. At the end of the reaction, the walls of both cylinders are covered with crystals of iodine or iodine monochloride. This experiment illustrates beautifully the diffusion of gases.

Cylinder of HI gas ; cylinder of Cl.

IODIC ACID

22. Preparation by the action of nitric acid on iodine. — When iodine is heated with fuming nitric acid, it is converted into iodic acid, with the liberation of the oxides of nitrogen.

Three grams of iodine are placed in a dry 200 cc. Erlenmeyer flask with 40 cc. of fuming nitric acid. The mixture is heated for a few minutes and the liquid poured off from the undissolved residue. On diluting with an equal volume of water and shaking with 10 cc. of carbon disulphide, the iodine is dissolved out, the solution becoming colorless. The carbon disulphide is drawn off in a separating-funnel, and the upper layer of liquid tested for iodic acid. The liquid is first filtered to free from drops of carbon disulphide. Starch paste is added to a portion of the solution and then a few drops of sulphur dioxide water. Iodine will be liberated and form the blue compound with starch. An excess of sulphurous acid causes the decolorization of the liquid.

$$3\ I_2 + 10\ HNO_3 = 6\ HIO_3 + 10\ NO + 2\ H_2O.$$

Separating-funnel ; I ; fuming HNO_3 ; CS_2 ; SO_2-water ; starch paste.

23. Decomposition by heat (iodic anhydride). — Iodic acid on heating in a porcelain evaporating dish loses water and forms the anhydride, iodic pentoxide. The heating should be regular and should cease at that point where iodine vapors are liberated. The molten mass is allowed to solidify and is then used in the following experiments on iodic anhydride.

Iodic anhydride, when heated in a test-tube, gives off oxygen, with the liberation of iodine. A glowing splinter introduced in the test-tube is immediately relighted.

The oxidizing power of iodic anhydride may be shown

by heating a mixture of the powdered anhydride and charcoal powder. The mixture on heating gives off violet vapors, the carbon feebly burning inside the tube.

A small quantity of flowers of sulphur or pulverized sugar is mixed with some powdered anhydride and heated in a dry test-tube. The reaction is very vigorous, being accompanied with a slight explosion.

$$2\ HIO_3 = I_2O_5 + H_2O.$$

$$2\ I_2O_5 = 2\ I_2 + 5\ O_2.$$

HIO_3; I_2O_5; powdered charcoal; S flowers; sugar.

24. Reduction by sulphurous acid. — Sulphurous acid acts in the same manner as hydriodic acid in reducing iodic acid. The reduction is first accompanied by a liberation of iodine. If, however, an excess of sulphurous acid is added, the iodine itself becomes converted to hydriodic acid.

If to a solution of iodic acid starch paste is added, no color will appear, as the combined iodine of the iodic acid does not produce the blue compound with starch. On the addition of a few drops of sulphurous acid the blue color will instantly appear. An excess of sulphurous acid discharges the color.

By adjusting the concentrations of the two solutions a most interesting experiment on the time required for a reaction to be completed may be made. The interaction of sulphurous acid and iodic acid appears to require some considerable time to begin, but having once started it proceeds instantaneously throughout the whole solution.

Ten grams of iodic acid are dissolved in a liter of distilled water and the solution bottled as a stock solution for use in this experiment. The solution is very permanent. Fifty cubic centimeters of water are saturated with sulphur dioxide by allowing the gas to bubble through it for a few

minutes. Twenty-five cubic centimeters of this saturated sulphurous acid solution are diluted to a liter and preserved in a bottle as a stock solution. This solution, however, as might be expected, is not so permanent as the iodic acid solution, hence it is advisable to make it fresh for every experiment. Two hundred and fifty cubic centimeters of water are placed in each of two clean beakers and to one 50 cc. of the stock iodic acid solution, and to the other 50 cc. of the stock sulphurous acid solution are added. A few drops of starch paste are then added to the dilute iodic acid solution. The contents of each beaker are then stirred to insure thorough mixing and then rapidly poured together in a large cylinder capable of holding 700 cc. The mixed liquids should be immediately stirred with a long, clean stirring rod. No apparent reaction will take place for about half a minute, when instantly the contents of the whole cylinder will be turned a deep blue. It is advisable to place the cylinder before a background of white paper.

The experiment can be made still more striking by using a metronome to indicate the seconds. By counting off the strokes of the metronome it will be easy to state beforehand on what stroke the color change will occur.

In determining before the lecture the length of time required for this reaction, it is essential to observe that no changes are made in the proportions of the ingredients, dilution, and temperature, since the length of time required for the reaction is greater at greater dilutions and with a higher content of iodic acid, while an increase in temperature shortens the time required for the reaction.

$$2\ HIO_3 + 5\ SO_2 + 4\ H_2O = 5\ H_2SO_4 + I_2.$$

Metronome ; HIO_3 ; SO_2-water (saturated) ; starch paste.

FLUORINE

HYDROFLUORIC ACID

1. Preparation from calcium fluoride and sulphuric acid.— Calcium fluoride, when warmed with concentrated sulphuric acid, undergoes decomposition, with the formation of hydrofluoric acid gas and calcium sulphate. The corrosive action of this gas on glass necessitates that the operation be carried out in a lead dish or a platinum crucible.

A thin paste of powdered fluorspar and concentrated sulphuric acid is placed in a lead dish or a platinum crucible and gently warmed. The gas is readily evolved and its etching action on glass is shown as follows:—

A piece of glass is carefully cleaned and coated with a thin layer of paraffin. By means of a sharp-pointed pin or needle a few characters are cut in the wax coating, care being taken to scratch clear through the wax down to the glass. The plate is then placed, with the waxed surface down, on top of the dish in which hydrofluoric acid is being generated and allowed to remain there for from five to twenty minutes. Care should be taken not to heat the dish sufficiently to melt the wax on the surface of the glass and thereby spoil the design.

In case a platinum crucible is used, it will be found difficult, owing to the small size of the opening, to etch a design of any size. The gas may be generated in the crucible,

however, and retained in a paper box of sufficient size to expose a much larger surface of glass to the action of the gas. A round pill box, some 8 to 10 cm. in diameter, has a hole cut in the bottom a little smaller than the maximum diameter of the platinum crucible (Fig. 61). The crucible is then crowded down into the hole until its rim is but a few millimeters above the bottom of the box.

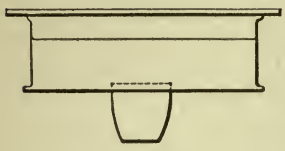

FIG. 61

The paste of calcium fluoride and concentrated sulphuric acid is very gently warmed in the platinum crucible, the gaseous fumes ascending into the pill box. A large glass plate may be prepared and etched as described above.

$$CaF_2 + H_2SO_4 = CaSO_4 + 2\ HF.$$

Lead dish or platinum crucible ; large pill box ; waxed glass plates ; CaF_2.

2. **Acidity.** — The gas formed in the preceding experiment will immediately redden a piece of moistened blue litmus paper.

3. **Etching glass with aqueous hydrofluoric acid.** — Aqueous hydrofluoric acid is obtainable in the market in rubber or paraffin bottles, and is much used in analytical operations. This aqueous solution etches glass fully as well as the gas.

A glass plate is coated with wax, prepared as described in Ex. 1, and a piece of filter-paper moistened with the aqueous hydrofluoric acid laid on top of the wax. In a few minutes the etching will be complete.

If a small rim of wax of sufficient height is made around the design, the aqueous acid may be poured directly upon the wax, and being retained by the rim will effect the etching as before.

In either of these experiments a paste of calcium fluoride and concentrated sulphuric acid may be substituted for the aqueous hydrofluoric acid.

Waxed glass plate ; aqueous HF ; CaF_2 (powdered).

4. Corrosive action of hydrofluoric acid on glass. — The corrosive action of aqueous hydrofluoric acid on glass and the consequent necessity of using bottles other than glass for holding this liquid may be shown by placing 50 cc. of the solution in a thin 200 cc. Erlenmeyer flask and gently warming. The flask should be set in an iron or, better, a platinum evaporating dish which is kept warm by a water-bath (Fig. 62). A rubber stopper fitted with a short glass elbow and a rubber tube conducts the fumes into the flue. In a short time (from one-half to three-quarters of an hour) the acid will have eaten through the bottom of the flask and run into the platinum evaporating dish.

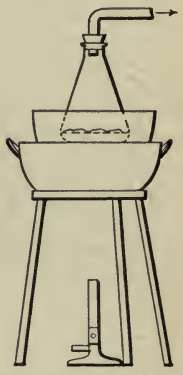

FIG. 62

Thin flask ; iron or platinum dish ; aqueous HF.

SULPHUR

SULPHUR

PROPERTIES

1. Roll sulphur. — Sulphur is a very poor conductor of both heat and electricity and, when gently warmed, at times even by the warmth of the hand, the roll will break in pieces.

A roll of sulphur is laid on a piece of asbestos paper and very gently warmed with a low flame. It will crackle and fall in pieces, owing to the unequal expansion produced by the heat.

Asbestos paper; roll of S.

2. Distillation of sulphur and preparation of sulphur flowers. — The vapors of sulphur, when allowed to escape into the air, condense in the form of a fine dust, the so-called flowers of sulphur.

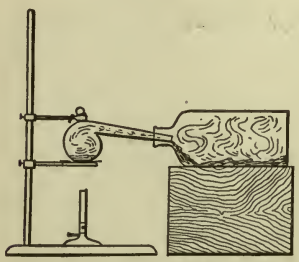

FIG. 63

A retort is one-third filled with roll sulphur, and the neck of the retort, which should be as short as possible, thrust in the mouth of a bottle, which is laid on its side and has a layer of asbestos paper, 10 cm. wide, along its length (Fig. 63). The sulphur is brought to a boil by means of a powerful burner and large quantities of the vapor are driven into the bot-

tle. An asbestos covering should be made for the mouth of the bottle and provided with a hole through which the neck of the retort may be thrust. A considerable portion of the sulphur vapor will condense in the neck of the retort and drop as a liquid upon the asbestos paper. The vapors will fill the bottle and be deposited all over the sides as a fine powder.

200 cc. retort; asbestos paper; bottle; large burner; roll S.

3. Preparation of plastic sulphur. — (*a*) Sulphur, when heated above its melting point, is changed into a black viscous mass having the properties of rubber.

Roll sulphur is gently heated in a 100 cc. Erlenmeyer flask and very carefully melted. A clear, yellow liquid results. If some of this light yellow liquid is poured into water, it will solidify to a hard, light yellow, opaque mass. On heating still further, however, the liquid becomes viscous and turns black, and the sulphur will not flow out, even if the flask is held mouth downwards.

If the liquid is heated still further, it again becomes more fluid, and can then be poured into cold water in a beaker. A funnel is placed mouth downwards in the beaker, and the thin stream of melted sulphur is poured around the stem of the funnel, forming a coil of solidified sulphur in the beaker (Fig. 64). On removing the sulphur from the beaker it will be found to be elastic and plastic.

(*b*) A 200 cc. glass-stoppered tubulated retort, preferably with a wide neck, is one-third filled with lumps of sulphur and clamped in such a position that it may be strongly heated in a large Bunsen burner. A sheet of asbestos paper is laid over the top of the retort, extending almost to the mouth, and held in place by having the stopper of the retort thrust through it. The contents of the retort are then melted and the heat gradually increased until they are vig-

orously boiling. The vapor partially condenses in the neck of the retort and falls in a thin stream into a large beaker of water, in which is immersed an inverted funnel about the diameter of the beaker (Fig. 64). The sulphur solidifies, and the beaker should be slowly revolved to allow the sulphur to collect in a spiral form on the funnel. It is advisable to ignite the sulphur vapor not condensed in the neck of the retort, as otherwise the flowers of sulphur will settle on the surface of the water and interfere with the proper cooling of the sulphur. When the contents of the retort are vigorously boiling, a flame of sulphur vapor, some 10 to 15 cm. in length, issues from the mouth of the retort. It is best to stop the heat before all the sulphur has been distilled. On withdrawing the funnel from the beaker the sulphur will be found as a bundle of fine threads and will possess elastic qualities.

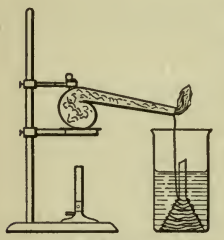

Fig. 64

200 cc. retort ; large burner ; beaker and funnel ; S.

4. Crystallization from fusion (prismatic sulphur). — A 150 cc. Jena glass beaker is two-thirds filled with roll sulphur, which is then carefully melted, care being taken to keep the temperature as low as possible. When the sulphur is completely melted, the flame is removed and the beaker imbedded in a dish of sand. As soon as it is cooled sufficiently to form a crust on the surface, a hole is made through the crust and the remaining melted sulphur poured out into water. A fine network of yellowish, transparent crystals will have formed all around the walls of the beaker, the needles extending into the centre of the mass.

5. Crystallization from carbon disulphide (octahedral sulphur). — Sulphur is quite soluble in carbon disulphide, and crystallizes from its solution in this solvent in octahedrons.

Twenty grams of pulverized roll sulphur are placed in a 100 cc. flask and covered with 50 cc. of carbon disulphide. The flask is then tightly closed with a cork (a rubber stopper cannot be used) and vigorously shaken. After a few moments the solution is allowed to stand, and the clear supernatant liquid decanted into a glass evaporating dish. As the liquid evaporates, the sulphur is deposited in octahedral crystals.

Glass evaporating dish ; roll S ; CS_2.

6. Union with iron powder. — Iron powder, when heated with twice its volume of sulphur flowers, combines with the latter with the evolution of heat and light to form ferrous sulphide. The mixture is gently heated in a hard-glass test-tube throughout the whole mass, and then the temperature is materially increased at the bottom. The union of the elements begins at this point and proceeds with a great evolution of heat and light through the contents of the test-tube.

A modification may be introduced by placing a small heap of the mixture of iron powder and sulphur flowers on a plate and touching it at one point with a hot glass rod. The mixture ignites, and the whole mass is soon converted to ferrous sulphide.

$$Fe + S = FeS.$$

Fe powder ; S flowers.

7. Union with iron at ordinary temperatures. — Iron powder and sulphur flowers unite at ordinary temperatures when mixed with a small quantity of water.

Twenty-two grams of iron powder and 15 g. of sulphur

flowers are rubbed together in a mortar with 7 cc. of water. After a thorough mixing the damp powder is placed on a white plate and moulded into a conical heap. The mixture is pressed firmly down with the fingers and allowed to stand some 15 to 20 minutes. Soon the mixture heats, and the cone cracks open and steam is seen to rise. The whole mass becomes black in color. That iron sulphide is actually formed is easily shown by adding some hydrochloric acid to a small quantity of the black powder and testing the hydrogen sulphide evolved.

Fe powder ; S flowers.

8. Combustion of iron in sulphur vapor. — Red-hot iron, when thrust into sulphur vapor, combines with the sulphur with incandescence, forming ferrous sulphide.

Sulphur is brought to a boil in a hard-glass test-tube and a piece of common iron wire gauze heated to redness in a Bunsen burner. On thrusting the gauze into the boiling sulphur the union takes place, the molten iron sulphide falling to the bottom of the test-tube.

9. Union with copper. — (*a*) Sulphur in a 100 cc. Jena glass flask is heated to boiling and the sulphur vapor allowed to burn at the mouth. A spiral of copper made by winding fine copper wire around a rod, or a piece of fine copper gauze is thrust into the neck of the flask through the flame of burning sulphur. The two elements unite with an evolution of light and heat.

$$Cu + S = CuS.$$

Fine copper wire or fine copper gauze ; roll S.

(*b*) A space, 1 cm. square, in a piece of thin sheet copper is heated strongly. When very hot a small bit of sulphur is thrown upon it. The sulphur and copper unite, forming cupric sulphide, which remains as a bright metallic black

spot in the centre of the sheet. The brittle nature of the product is shown by thrusting the point of a lead pencil through the spot.

Cu sheet; S.

10. Combustion of magnesium in sulphur vapor. — Burning magnesium, when lowered into sulphur vapor, continues to burn with the formation of magnesium sulphide.

Sulphur is brought to a boil in a 100 cc. flask and a 15 cm. strip of magnesium ribbon is ignited in the air. When the magnesium is burning freely, it is lowered through the mouth of the flask into the sulphur vapor, where it continues to burn with the formation of magnesium sulphide.

$$Mg + S = MgS.$$

Boiling S; Mg ribbon.

11. Combustion of zinc and sulphur. — A mixture of two parts of zinc and one part of sulphur flowers, when thrown into a hot iron dish or saucer, burns violently, forming zinc sulphide.

This mixture is also readily ignited by touching it with a hot glass rod. A small heap of the mixed powders is placed on a brick or a piece of asbestos paper and touched with a glass rod which has been heated in the Bunsen flame.

$$Zn + S = ZnS.$$

Iron dish or crucible; Zn dust; S flowers.

HYDROGEN SULPHIDE

PREPARATION

12. By the union of hydrogen and sulphur. — Hydrogen, when passed over sulphur in the cold, has no action on the element, though, if the sulphur is heated, a portion of it combines with the hydrogen to form hydrogen sulphide.

A small lump of sulphur is placed in a bulb-tube through which dry hydrogen is conducted. An elbow attached to the end of the bulb-tube dips into lead acetate solution. No change is observed in the lead acetate solution until the sulphur is heated, when, almost immediately, the presence of hydrogen sulphide is shown in the issuing gas by the formation of a black precipitate of lead sulphide in the solution.

Instead of using the bulb-tube, the sulphur may be placed in a dry test-tube, fitted with a two-holed cork. Hydrogen is conducted through a glass elbow in the cork extending to the bottom of the test-tube, and issues through a glass elbow in the other hole of the cork. A paper moistened with lead acetate solution held in the issuing gas remains unchanged until the sulphur is warmed, when it is immediately blackened by the hydrogen sulphide formed.

$$H_2 + S = H_2S.$$

Bulb-tube; H generator; S.

13. From the reduction of sulphur dioxide by means of hydrogen in the presence of platinized asbestos. — Sulphur dioxide, when mixed with hydrogen and passed over heated platinized asbestos, is reduced to sulphur, which is deposited in the tube. The excess of hydrogen reacting on the heated sulphur produces a small amount of hydrogen sulphide, which may be tested in the issuing gas.

The apparatus (Fig. 69, p. 159) is adapted for this purpose, hydrogen being substituted for the oxygen. The hydrogen and sulphur dioxide bubbling through the sulphuric acid mix and pass out through the third neck into the bulb-tube containing platinized asbestos. Until the platinum is heated no reaction takes place, but on warming the bulb the platinized asbestos will be seen to glow, and a deposit of sulphur will appear in the farther end of the

tube. The issuing gas, when tested with a piece of paper moistened with lead acetate solution, shows the presence of hydrogen sulphide.

3-necked Wolff bottle; H generator; SO_2 generator; platinized asbestos.

14. From ferrous sulphide and hydrochloric or sulphuric acid. — Ferrous sulphide, when treated with dilute sulphuric acid, or, better, hydrochloric acid, liberates large quantities of hydrogen sulphide. Though the gas generated by this operation is not perfectly pure, containing, as it does, considerable quantities of hydrogen, it is used, nevertheless, for almost all analytical operations, as it is an indispensable adjunct in analytical chemistry. Unfortunately the evil-smelling and poisonous properties of the gas necessitate special precautions in its preparation, collection, and use.

The gas is readily prepared by placing a few lumps of ferrous sulphide in the gas generator (Ex. 7, p. 43). Dilute sulphuric acid (1 volume of concentrated acid to 14 of water), or, better, dilute hydrochloric acid (1 volume of concentrated acid to 1 of water), is poured through the thistle-tube. The reaction begins almost immediately. The gas should be dried by means of calcium chloride only, as it is decomposed by sulphuric acid.

In case but a small quantity of the gas is desired, the apparatus as above described is satisfactory, though it is necessary to prevent any quantity of the gas from escaping into the room. This can best be accomplished by inserting after the wash-bottle a three-way stop-cock, such as is shown in Fig. 39, p. 81. One arm of the stop-cock is connected with the flue or with some suitable absorbing bottle, the other directly with the apparatus into which the hydrogen sulphide is to be conducted.

Innumerable devices for the evolution of hydrogen sul-

phide have been described in chemical journals, but as yet no ideal portable apparatus for supplying a constant stream of this gas has been devised. The simplest form is probably the Kipp generator, the middle bulb being filled with lumps of ferrous sulphide as large as can conveniently pass through the tubulature, while the acid chamber is filled with sulphuric or hydrochloric acid diluted as described above. The replenishment of this apparatus with acid and ferrous sulphide is, however, a very disagreeable task, and consequently a great drawback to its general use.

In most laboratories large quantities of hydrogen sulphide are prepared for use with classes in qualitative and quantitative analysis. The ideal arrangement is that in which distilled water is supercharged with the gas and retained in tin-lined steel cylinders. This method is the one adopted in the chemical laboratory of Harvard College. The number of forms of apparatus for preparing the gas on the small scale is only exceeded by the number of devices for preparing it on a large scale for classes of students. While each has its merits, a description is given in the Appendix (p. 420), of the apparatus used in Wesleyan University, which for regularity, simplicity, and economy is not excelled by any other form known to the writer.

For lecture-table purposes the arrangement for obtaining hydrogen sulphide is to conduct a separate pipe (preferably a quarter-inch lead pipe) from the large generator to a glass stop-cock securely fastened to a partition under the lecture table. A second tube is then connected with the glass stop-cock, which conducts the gas to a metal cock on the top of the table, where by simply turning the cock a full supply of the gas is instantly secured. The glass stop-cock is used to cut off the gas when not in use, and is preferable to a metal cock on account of its non-corrosive character. During the lecture the metal stop-cock on the upper part of the desk

may be used for regulating the supply of gas, though the supply should be cut off with the glass stop-cock at the end of the lecture. This arrangement can be applied to many of the existing forms of hydrogen sulphide generators, and is by far the most satisfactory method of obtaining this gas for lecture table purposes.

$$FeS + 2\,HCl = FeCl_2 + H_2S.$$

$$FeS + H_2SO_4 = FeSO_4 + H_2S.$$

Gas generator (Ex. 7, p. 81) ; three-way cock ; FeS.

15. By the action of hydrochloric acid on antimony sulphide. —Native antimonious sulphide (stibnite) yields, when treated with hydrochloric acid, a very pure form of hydrogen sulphide uncontaminated with free hydrogen.

A few grams of the pulverized mineral are placed in a 300 cc. Erlenmeyer flask fitted with a dropping-funnel and elbow (Fig. 3, p. 11). Concentrated hydrochloric acid is allowed to drop into the flask, which is gently warmed. A steady evolution of pure hydrogen sulphide is obtained.

A measured volume of this gas will be almost completely absorbed by sodium hydroxide, as shown in Ex. 18.

$$Sb_2S_3 + 6\,HCl = 2\,SbCl_3 + 3\,H_2S.$$

300 cc. Erlenmeyer flask ; dropping-funnel ; eudiometer (Fig. 11, p. 26) ; stibnite.

PROPERTIES

16. Collection of the gas. — (*a*) In many experiments it is necessary to use dry hydrogen sulphide in dry containers. As this gas is somewhat heavier than air, it can be collected by displacement, though provision should be made to conduct the excess of the gas into the flue or some proper absorber.

A cylinder is fitted with a two-holed rubber stopper. A glass tube extends to the bottom of the cylinder through one

of the holes in the cork, and through the other hole a glass elbow is thrust. The hydrogen sulphide is conducted through the long tube to the bottom of the jar, where it collects and forces the air out through the elbow at the top. When the jar is full, the cork is rapidly withdrawn and a glass plate slipped over the mouth of the jar. The cork should be immediately thrust into a second cylinder.

The acid nature of the gas, as well as its aqueous solution, should be shown.

(*b*) Owing to its solubility in cold water, it is essential when collecting this gas over water, to have the water as warm as possible, and to use only a porcelain or glass dish as a pneumatic trough. The gas is much less soluble in salt solution than in water, and may accordingly be collected over brine.

Porcelain or glass pneumatic trough; H_2S generator; hot water; sat. sol. NaCl.

17. Solubility in water. — (*a*) Hydrogen sulphide is readily absorbed by water.

A eudiometer tube is two-thirds filled with hydrogen sulphide, the thumb placed over the mouth of the tube and the tube shaken thoroughly. The gas will dissolve in the water remaining in the tube, producing a suction, and if the tube is opened under water, the water will rise inside. The current of hydrogen sulphide used to fill the tube should be very rapid, so as to prevent the water from being saturated with the gas while the tube is being filled.

(*b*) The eudiometer (Fig. 11, p. 26) may be used in this experiment, in which case it is filled with hydrogen sulphide over water and ice-cold water allowed to flow down the inside of the tube into the gas. As the gas is absorbed, the level of the water in the tube rises.

H_2S generator; eudiometer (Fig. 11, p. 26); ice-water.

(*c*) A flask is rapidly filled with hydrogen sulphide over water until half of the water in the flask has been driven out. The palm of the hand is then placed on the mouth of the flask, which is thoroughly shaken. The gas is absorbed by the water, producing a suction capable of supporting the flask in a hanging position in the palm of the hand.

In case the water in the flask becomes saturated with the gas, which is almost sure to happen if the current is not rapid, the operation is best performed by filling the flask completely with the gas and then pouring in one-third of its volume of ice-cold water. On covering the flask with the moistened palm and shaking it, a very strong suction will be obtained.

Stout-walled flask; H_2S generator; ice-water.

(*d*) A saturated solution of the gas may be obtained by using the apparatus, Fig. 85, p. 196.

18. Solubility in sodium hydroxide. — The solubility of hydrogen sulphide in sodium hydroxide is shown by filling the eudiometer (Fig. 11, p. 26) with hydrogen sulphide over a saturated solution of sodium chloride. Sodium hydroxide is allowed to flow through the stop-cock into the gas, which will immediately be absorbed and the liquid will rise inside the tube.

If the hydrogen sulphide is prepared from commercial ferrous sulphide, considerable hydrogen gas will be present in the evolved gas and therefore be unabsorbed by the sodium hydroxide.

If the hydrogen sulphide is prepared from the action of hydrochloric acid on antimony sulphide, the gas will be much more pure, and but a small quantity of gas will remain unabsorbed, *i.e.*, less than half a cubic centimeter out of 100 cc. of hydrogen sulphide.

19. Combustion in air. — Hydrogen sulphide, though a non-supporter of combustion, burns with a blue flame.

A lighted candle lowered into a jar containing hydrogen sulphide is extinguished, and the hydrogen sulphide ignited. The gas burns at the mouth of the jar, but owing to the deficiency of oxygen in the lower portions of the jar, the sulphur is deposited instead of being oxidized as fast as it is liberated, hence the walls of the jar become covered with a thin film of flowers of sulphur.

To demonstrate especially this latter phenomenon, it is advisable to have a tall jar, as this form retards the entrance of the air.

Tall jar of H_2S ; candle on wire.

20. Deposition of sulphur in the incomplete combustion of hydrogen sulphide. — If air is burned in an atmosphere of hydrogen sulphide, the heat from the flame will be sufficient to cause a decomposition of the surrounding gas with an accompanying deposition of sulphur.

The apparatus with which this flame of air is produced, is that shown in Fig. 142, p. 341. A strong current of hydrogen sulphide is conducted through the elbow in the bottom of the lamp chimney and is ignited at the orifice at the top. A gentle current of air, best obtained from the water blast, is passed through the long tube, which may be pushed up through the hole in the cork into the centre of the hydrogen sulphide flame at the top of the lamp chimney. On slowly lowering the tube, the air will be seen to be burning in the atmosphere of hydrogen sulphide, and a deposit of sulphur will be obtained on the walls of the chimney.

Lamp chimney and fittings (Fig. 142, p. 341) ; H_2S generator ; air-blast or gasometer.

21. Decomposition in a hot tube. — Dry hydrogen sulphide, when passed through a glass tube which is strongly heated

with a Bunsen burner, is decomposed, and sulphur is deposited on the cooler portions of the tube.

$$H_2S = H_2 + S.$$

22. Explosion of a mixture of hydrogen sulphide and oxygen. — A round-bottomed ginger-ale bottle is filled with three volumes of oxygen and two volumes of hydrogen sulphide. The mixture is well shaken, and then a flame held at the mouth of the bottle.

$$2\,H_2S + 3\,O_2 = 2\,H_2O + 2\,SO_2.$$

23. Action on sulphur dioxide. — (*a*) In the presence of a small amount of moisture, hydrogen sulphide reacts with sulphur dioxide, with the liberation of sulphur. This reaction is of especial interest in indicating the deposition of sulphur in volcanoes. The operation may be carried out in the simplest manner by inverting a cylinder of hydrogen sulphide collected over water, mouth downwards, on top of a cylinder of equal size, containing sulphur dioxide. When the glass plates are slipped from between the two cylinders, the gases unite with a heavy deposition of sulphur on the walls of the cylinders. As the sulphur dioxide is nearly twice as heavy as the hydrogen sulphide, the diffusion of the two gases is somewhat slow, and gives an excellent illustration of this phenomenon. In case it is desired to hasten the action, the cylinders are shaken.

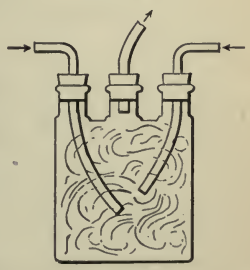

Fig. 65

The reaction may be shown on a much more extended scale by conducting the two gases from suitable generators into a large three-necked Wolff bottle (Fig. 65). Hydrogen sulphide is conducted through one neck of the

Wolff bottle, and sulphur dioxide through another. The third neck is fitted with a cork, carrying a glass elbow connected with the flue. After a few minutes, the whole interior of the Wolff bottle becomes coated with a layer of finely divided sulphur.

The bottle should be cleaned immediately after use, as the sulphur, if allowed to dry on the walls of the bottle, is very hard to remove. The reaction is not altogether as simple as is given in Equation (1) below, as a certain small quantity of pentathionic acid is formed.

$$(1) \quad SO_2 + 2\,H_2S = 2\,H_2O + 3\,S.$$

$$(2) \quad 5\,SO_2 + 5\,H_2S = H_2S_5O_6 + 4\,H_2O + 5\,S.$$

Wolff bottle (3-necked) ; H_2S generator ; SO_2 generator ; cylinder of H_2S ; cylinder of SO_2.

(*b*) The interaction of hydrogen sulphide and sulphur dioxide may also be carried out in aqueous solution. Aqueous solutions of the two gases, when mixed, produce a heavy precipitate of sulphur.

By conducting hydrogen sulphide into a solution of sulphur dioxide, or by conducting sulphur dioxide into a solution of hydrogen sulphide, the precipitate of sulphur is also obtained.

24. Combustion of iron. — A bundle of iron wires, or better, a bit of steel wool (Ex. 16, p. 23), when held in the flame of burning hydrogen sulphide, will take fire in the gas and burn, forming ferrous sulphide.

H_2S supply ; steel wool.

25. Ignition by fuming nitric acid. — Strong oxidizing agents, such as fuming nitric acid, effect the rapid oxidation and subsequent ignition of hydrogen sulphide.

A 300 cc. cylinder is filled with hydrogen sulphide,

covered with a ground-glass plate, and placed mouth upwards on the table. A few cubic centimeters of fuming nitric acid are gently warmed and poured into the jar of the gas. The hydrogen sulphide is ignited, with a slight explosion, and care should be taken to avoid danger from the spurting out of drops of acid. Dense fumes of nitrogen oxides are evolved, and sulphur is deposited on the walls of the cylinder.

300 cc. cylinder of H_2S; fuming HNO_3.

26. Decomposition by sodium. — Hydrogen sulphide, when passed over heated sodium, is decomposed, the sulphur uniting with the sodium to form sodium sulphide, and the hydrogen escaping from the tube.

A few small pieces of sodium, which should be dried and freed from crust, are placed in a hard-glass bulb-tube through which a current of hydrogen sulphide is being passed. On heating the metal gently it takes fire, glowing brightly, and the issuing hydrogen, when ignited, burns with a yellow sodium flame. The yellow residue in the bulb consists of sodium sulphide, which may be dissolved out with water and tested.

$$H_2S + 2\,Na = Na_2S + H_2.$$

Bulb-tube; H_2S supply; Na.

27. Action on solutions of metallic salts. — The great importance of the use of hydrogen sulphide in analytical chemistry renders a study of its action on solutions of metallic salts essential. The simplest method of showing its action is by adding small portions of an aqueous solution of hydrogen sulphide to solutions of the various metals in small cylinders. The following selection of metals shows the great variety of color in the sulphides and the necessity of an alkaline solution to effect the precipitation of the sul-

phide from certain salt solutions. The cylinders should contain respectively solutions of cupric sulphate, antimonious chloride, cadmium chloride, lead nitrate, zinc sulphate, manganese sulphate, and cobalt nitrate. In preparing the solution of antimonious chloride the addition of tartaric acid will prevent the precipitation of the white oxychloride. The solutions of zinc, manganese, and cobalt give no precipitate with hydrogen sulphide, though on the addition of a small quantity of ammonium hydroxide the sulphides are immediately precipitated.

The operation may also be carried out by passing hydrogen sulphide through the various salt solutions contained in gas washing-bottles. The hydrogen sulphide generator used in this experiment must have a very long safety-tube to permit an increase of pressure within the generator sufficient to cause the gas to bubble through the successive liquids. To hasten the operation, dilute solutions of the salts are used. The cylinders are connected in series in the order above indicated. In case a Kipp generator is used to furnish the hydrogen sulphide, the necessary pressure is obtained by inserting a cork carrying a short glass tube, a rubber tube, and a pinch-cock into the opening in the top of the generator. By closing the pinch-cock when the generator is in operation the acid is prevented from rising into the acid reservoir, and consequently a greater pressure is obtained. The rubber tube and pinch-cock may be replaced by a safety-funnel containing mercury.

$$CuSO_4 + H_2S = CuS + H_2SO_4.$$

$$2\,SbCl_3 + 3\,H_2S = Sb_2S_3 + 6\,HCl.$$

etc. etc.

Seven gas washing-bottles; H_2S generator, with extra long thistle or Kipp H_2S generator; solutions of $CuSO_4$, $SbCl_3$, $CdCl_2$, $Pb(NO_3)_2$, $ZnSO_4$, $MnSO_4$, $Co(NO_3)_2$.

HYDROGEN PERSULPHIDE

28. Preparation by the action of hydrochloric acid on calcium polysulphide. — Calcium polysulphide is made by adding 40 g. of sulphur flowers to 300 cc. of water, to which 20 g. of quicklime have been added. The mixture is boiled ten minutes, and then allowed to settle. The cooled liquid is decanted off and poured in a thin stream into a mixture of 100 cc. of hydrochloric acid and 50 cc. of water, in a liter separating-funnel, which is shaken during the addition. After a few minutes the greater portion of the hydrogen persulphide formed will have settled to the bottom of the funnel as a heavy, yellowish oil. The oil is drawn off into a small flask containing fused calcium chloride, where it is dried.

$$CaS_5 + 2\,HCl = H_2S_2 + CaCl_2 + 3\,S. \quad [?]$$

Liter separating-funnel ; small flask ; CaO ; S flowers ; fused $CaCl_2$.

29. Solubility in carbon disulphide. — Hydrogen persulphide is mixed in a test-tube with two or three times its volume of carbon disulphide. The two liquids are perfectly miscible, and the solution remains undecomposed much longer than the pure hydrogen persulphide.

H_2S_2 ; CS_2.

30. Bleaching action. — Very dilute litmus solution, when added to two drops of hydrogen persulphide in a test-tube, is bleached. The reaction is not immediate, and follows only after vigorous shaking. Sulphur is precipitated.

31. Decomposition in an alkaline solution. — Hydrogen sulphide, like hydrogen peroxide, is more stable in an acid than in an alkaline solution. The compound decomposes in a dilute alkaline solution, setting free sulphur and forming hydrogen sulphide.

Fifty cubic centimeters of water are added to 2 drops of hydrogen persulphide in a 100 cc. flask. One drop of sodium hydroxide solution is added, and the mixture vigorously shaken. Almost immediately, even in the cold, the liquid becomes turbid from sulphur precipitated, and on warming the solution, hydrogen sulphide is expelled.

$$H_2S_2 = H_2S + S.$$

32. Reduction of silver oxide. — Hydrogen persulphide, like hydrogen peroxide, reduces silver oxide. A few centigrams of silver oxide are allowed to fall upon 2 drops of hydrogen persulphide in a dry test-tube. The reaction is vigorous.

SULPHUR MONOCHLORIDE

33. Preparation by the action of chlorine on sulphur. — Dry chlorine acts on sulphur with the production of a yellow, oily liquid, sulphur monochloride.

A 400 cc. tubulated retort is one-third filled with flowers of sulphur. Dry chlorine is conducted through a glass elbow inserted in the tubulature, and the neck of the retort is thrust into a filter bottle, whose side tube is connected with a draft (Fig. 66). Provision should be made for heating the sulphur and cooling the receiver. The sulphur is strongly heated, and the chlorine current maintained at a rapid rate. The tube conducting the chlorine should be lowered so as to almost touch the surface of the melted sulphur. After a few cubic centimeters

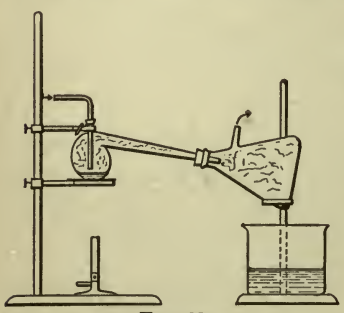

FIG. 66

of the chloride have collected in the receiver the operation should be stopped.

$$2\ S + Cl_2 = S_2Cl_2.$$

400 cc. tubulated retort; filter-flask; S flowers; Cl generator.

34. Solubility of sulphur in sulphur monochloride. — A few pieces of sulphur are placed in a test-tube and covered with sulphur monochloride. By shaking, or more rapidly by gentle boiling, the sulphur is completely dissolved.

35. Decomposition by water. — Sulphur monochloride, when allowed to stand in contact with water, is gradually decomposed with the liberation of sulphur and hydrogen sulphide. One cubic centimeter of sulphur monochloride is placed in a test-tube with 2 or 3 cc. of water and vigorously shaken. The decomposition is rapid and the contents of the tube become warm.

SULPHUR DIOXIDE AND SULPHUROUS ACID

PREPARATION

36. From sulphur and oxygen. — Sulphur burns in the air or in pure oxygen to form sulphur dioxide.

A 40 cm. length of combustion-tube, provided with a cork at each end, is clamped in a horizontal position, and a wad of asbestos or glass wool placed at one end of the tube. A boat, in which a few pieces of roll sulphur are placed, is inserted in the other end of the tube, and a gentle stream of oxygen is admitted, driving out all air. The cork is withdrawn, the sulphur ignited, and the current of oxygen continued through the tube. The sulphur burns with a blue flame, forming sulphur dioxide, which escapes at the other

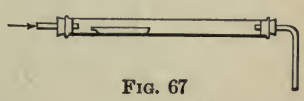

FIG. 67

end of the combustion-tube. The plug of asbestos serves to retain any unburned sulphur which may distil over. The issuing gas may be collected in cylinders by displacement and used in any of the experiments described beyond.

40 cm. length combustion-tube (Fig. 67); porcelain boat; glass wool or fibrous asbestos; S; O supply.

37. Volumetric relation of the sulphur dioxide formed to the oxygen used. — Sulphur, when burned in an atmosphere of oxygen, yields one volume of sulphur dioxide for each volume of oxygen consumed, hence in burning sulphur in a confined volume of oxygen the volume of the product remains the same as the volume of the oxygen used.

A 700 cc. Jena glass distillation flask is filled with dry oxygen by displacement, and the arm connected with one limb of a U-tube half filled with mercury (Fig. 68). The cork is provided with a glass rod carrying a small platinum deflagrating-spoon at the end, in which sulphur is burned. The deflagrating-spoon is easily made from a small piece of platinum foil which is fastened to the glass rod by means of platinum wire. A 4 mm. piece of roll sulphur is placed in the platinum spoon, and after ignition in the air, is immediately thrust into the flask, where it continues to burn with great brilliancy. The cork must be firmly inserted in the neck of the flask, and the union between the U-tube and the flask must be very tight. As the sulphur burns, the gases expand, and the mercury rises in the open arm of the U-tube. On cooling, the gases contract, and at the end of the

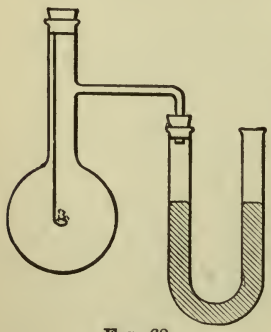

Fig. 68

operation the level in the arms of the U-tube will be found to be the same.

700 cc. Jena glass distillation flask; 1-holed rubber stopper; U-tube; platinum deflagrating-spoon; Hg; S.

38. From sulphuric acid and copper. — Sulphuric acid, when heated with copper, becomes reduced, liberating sulphur dioxide. This reaction was formerly used to prepare the gas on the lecture table, but the method of Experiment 41 is preferable.

The gas may be prepared on the small scale by heating a few grams of copper clippings with concentrated sulphuric acid in a 100 cc. Erlenmeyer Jena glass flask.

$$Cu + 2\ H_2SO_4 = CuSO_4 + 2\ H_2O + SO_2.$$

100 cc. Erlenmeyer flask; Cu clippings.

39. From sulphuric acid and powdered charcoal. — Charcoal reduces sulphuric acid, with the formation of varying amounts of sulphur dioxide, carbon monoxide and dioxide.

The impure gas may be made by heating a thin paste of powdered charcoal and concentrated sulphuric acid in a 100 cc. Jena glass Erlenmeyer flask. The reaction requires considerable heat to start, but after a short time sulphur dioxide may be detected at the mouth of the flask.

$$4\ H_2SO_4 + 3\ C = 4\ H_2O + 4\ SO_2 + CO_2 + 2\ CO.$$

100 cc. Jena glass Erlenmeyer flask; powdered charcoal.

40. From sulphuric acid and sulphur. — Sulphur reacts with hot concentrated sulphuric acid to form sulphur dioxide.

A thin paste of sulphur flowers and concentrated sulphuric acid is heated in a 100 cc. Jena glass Erlenmeyer flask. As the temperature of the sulphuric acid rises almost to the boiling point, the reaction begins. The

sulphur melts to a globule, and thus presents but little surface for the action of the acid, hence the quantity of sulphur dioxide formed is very small.

$$2\,H_2SO_4 + S = 2\,H_2O + 3\,SO_2.$$

100 cc. Jena glass Erlenmeyer flask; S flowers.

41. By the action of sulphuric acid on sodium acid sulphite. — By far the most satisfactory method for the preparation of sulphur dioxide is that in which the reaction between sulphuric acid and sodium acid sulphite is used. This latter compound can be readily obtained in the market at a very low price.

Sodium acid sulphite is placed in the bottom of the 300 cc. Erlenmeyer flask of the apparatus (Fig. 45, p. 92). The dropping-funnel is filled with dilute sulphuric acid, made by pouring one volume of concentrated sulphuric acid into an equal volume of water and cooling the mixture. The glass elbow is connected with a gas washing-bottle containing concentrated sulphuric acid, and the dry gas is collected by displacement in two or three empty cylinders. On allowing the dilute sulphuric acid to drop slowly upon the sodium acid sulphite, a very regular evolution of sulphur dioxide is obtained.

$$2\,NaHSO_3 + H_2SO_4 = Na_2SO_4 + 2\,SO_2 + 2\,H_2O.$$

300 cc. Erlenmeyer flask; dropping-funnel; $NaHSO_3$; dil. H_2SO_4 (1:1).

PROPERTIES

42. Liquefaction of sulphur dioxide. — Pure dry sulphur dioxide, when passed through a tube immersed in a freezing mixture, condenses to a colorless liquid.

Sulphur dioxide generated according to the method of the preceding experiment is first dried by passing through a gas washing-bottle containing sulphuric acid, and then

conducted into a U-tube immersed in salt and ice. The gas is there liquefied. Many expensive tubes in which to condense the gas are on the market, though, unless large quantities of the liquid sulphur dioxide are required, the operation is well shown by the ordinary U-tube.

The liquefied gas is readily obtained in small tin cylinders at a low price, and is best used in this form for the experiments on the production of cold by its evaporation. The cans are cooled in a mixture of salt and ice before being opened, and if they are kept immersed in the freezing mixture, there will be no great loss by evaporation.

U-tube; ice and salt; SO_2 generator.

43. Freezing action of liquefied sulphur dioxide. — (*a*) Liquid sulphur dioxide, when allowed to evaporate spontaneously, produces an intense cold which may be utilized to freeze water.

Five cubic centimeters of liquid sulphur dioxide are poured on 10 cc. of water in a small beaker. The mixture is stirred and immediately solidifies to a crystalline mass. Provision should be made for the escape, into the flue or hood, of the vapors of sulphur dioxide.

(*b*) A few drops of water are placed on a block of wood and a small beaker set on the wet portion. On pouring 5 cc. of liquefied sulphur dioxide into the beaker, the liquefied gas evaporates, producing an intense cold. The water under the beaker freezing causes the wood to adhere to the beaker.

Block of wood; liquefied SO_2.

44. Production of the Leidenfrost phenomenon with liquefied sulphur dioxide. — Liquefied sulphur dioxide, when thrown into a hot platinum dish, assumes the spheroidal state, giving a striking illustration of the Leidenfrost

phenomenon of a liquid, which boils below zero, remaining in the liquid form in a red-hot dish.

A platinum dish is brought to a red heat in a powerful Bunsen lamp and 10 cc. of liquefied sulphur dioxide poured into it. Instantly the liquid assumes the spheroidal form, and may be poured out of the dish upon a plate.

The experiment may be made still more striking by following the introduction of the liquid sulphur dioxide by an immediate addition of 10 cc. of water to the contents of the platinum dish. If the dish is then quickly removed by means of crucible tongs and the contents thrown on a plate, it will be seen that sufficient sulphur dioxide has remained unvolatilized to freeze the water, which will remain in the plate in the form of ice.

Platinum dish; plate; liquefied SO_2.

45. Specific gravity of gaseous sulphur dioxide. — Sulphur dioxide is twice as heavy as air, and when a liter of the gas is poured into a beaker on a balance arm, as in Ex. 9, p. 47, a marked deflection of the pointer is obtained.

If a liter of the gas is poured upon the paper wheel described in Ex. 36, p. 313, it may be made to rotate.

Lecture-balance; paper wheel (Fig. 125, p. 313); liter cylinder of SO_2.

46. Action on litmus. — A moistened strip of blue litmus paper is thrust into a jar of the gas, where, owing to the acid nature of the sulphur dioxide, it is instantly turned red.

47. Action on potassium dichromate solution. — Five cubic centimeters of potassium dichromate solution, when poured into the gas, are immediately turned green, owing to the reducing action of the sulphur dioxide.

48. Solubility in water. — (*a*) If a small piece of ice or a few drops of water are allowed to enter a tube of the gas collected over mercury, the absorption is very rapid, the mercury rising to take the place of the absorbed gas.

(*b*) A cylinder filled with sulphur dioxide is opened mouth downwards under water. The gas is absorbed and the water rises and completely fills the cylinder.

SO_2 in tube collected over mercury; ice.

49. The gas does not support the combustion of a candle. — A lighted candle or a burning taper, when lowered into the gas, is immediately extinguished.

50. Bleaching action. — Sulphur dioxide is a powerful bleaching agent and is much used for this purpose in technical chemistry. Its bleaching action is best shown by decolorizing a red rose with the gas. The rose is lowered into a jar of sulphur dioxide and allowed to remain there some minutes. The color will rapidly disappear. The color may be restored by holding the flower in chlorine or in the vapor of fuming nitric acid. The restoration of the color depends upon the fact that the sulphur dioxide is oxidized to sulphuric acid by chlorine and fuming nitric acid.

SO_2 gas; jar of Cl; fuming HNO_3; red rose.

51. Restoration of the color to a rose bleached by sulphur dioxide. — The colorless compound formed by the union of sulphur dioxide with the coloring matter of the flower is destroyed by the action of sulphuric acid, hence by immersing the rose bleached in the preceding experiment in 50 per cent sulphuric acid the color is restored. The acid should be prepared and cooled before use.

Rose from preceding experiment; H_2SO_4 (50 per cent).

52. Combustion of iron or tin. — Sulphur dioxide, while not supporting the combustion of carbonaceous material, will support the combustion of certain of the metals.

A small quantity of iron or tin powder is placed in a bulb-tube through which a gentle stream of sulphur dioxide is conducted. On heating the iron powder it glows, with the formation of iron sulphide and oxide.

Bulb-tube; SO_2 generator; Fe powder; powdered Sn.

53. Union of sulphur dioxide and lead dioxide. — Sulphur dioxide and lead dioxide unite directly to form lead sulphate.

A small quantity of well-dried lead dioxide is placed on a piece of asbestos paper covering the bowl of a deflagrating-spoon. On lowering the spoon into a jar of dry sulphur dioxide the dark-colored oxide is converted to white lead sulphate, the change being accompanied by a strong glowing.

The union may also be accomplished by conducting a stream of sulphur dioxide through a bulb-tube containing a quantity of the lead dioxide.

$$PbO_2 + SO_2 = PbSO_4.$$

Bulb-tube; deflagrating-spoon; asbestos paper; SO_2 supply; jar of SO_2; PbO_2.

54. Reduction of potassium permanganate solution. — The intense color of the solution of potassium permanganate is immediately discharged by adding sulphur dioxide water to it in a cylinder.

The experiment can be varied by allowing the deep-colored permanganate solution to flow from a burette into a cylinder of the sulphur dioxide solution. As the permanganate drops into the sulphurous acid its color vanishes until all the sulphurous acid has been oxidized, when one

more drop of the permanganate will permanently color the whole solution. The permanganate solution should in each case be acidified with sulphuric acid to prevent the precipitation of oxides of manganese.

Burette ; $KMnO_4$ solution ; SO_2 water.

55. Electrolysis of sulphurous acid. — Nascent oxygen and hydrogen react with sulphurous acid, forming in the first case sulphuric acid, and in the second reducing the sulphurous acid to sulphur.

A 10 per cent solution of sulphuric acid is saturated with sulphur dioxide and the liquid placed in the electrolytic apparatus (Fig. 46, p. 95). On passing a current from a bichromate battery through the platinum electrodes, it will be found that very little, if any, gas collects in the tubes. The oxygen liberated at the positive pole is used up in oxidizing the sulphurous acid to sulphuric acid, while the hydrogen formed at the negative pole immediately effects the reduction of the sulphurous acid to sulphur. The liquid around the negative pole becomes milky from the liberation of free sulphur.

Electrolytic apparatus (Fig. 46, p. 95) ; Pt electrodes ; bichromate battery ; 10 per cent H_2SO_4 saturated with SO_2.

HYDROSULPHUROUS ACID

56. Preparation. — Sulphur dioxide water or sulphurous acid in the presence of finely divided zinc is reduced to hydro- or hyposulphurous acid.

A 100 cc. Erlenmeyer flask is half filled with a saturated solution of sulphur dioxide in water and a few grams of pulverized zinc are added. The solution, which contains free hydrosulphurous acid, becomes somewhat yellowish, and a portion of the zinc is dissolved. As the reaction continues,

the acid is decomposed and a small quantity of sulphur is precipitated.

$$H_2SO_3 + H_2 = H_2SO_2 + H_2O.$$

SO_2 water; granulated Zn.

57. Bleaching action. — Indigo solution, which is not bleached by sulphurous acid, is readily bleached by hydrosulphurous acid. A small quantity of indigo solution is placed in each of two cylinders. Sulphurous acid is added to the first, and hydrosulphurous acid to the second. The latter only is bleached. Litmus paper is similarly bleached.

H_2SO_2; indigo solution.

58. Reducing action. — Hydrosulphurous acid possesses strong reducing properties and precipitates the metals mercury and silver from solutions of their soluble salts. The reactions may be carried out in test-tubes by adding a small quantity of hydrosulphurous acid to the respective salt solutions.

The reaction with cupric sulphate proceeds in two stages. A small quantity of the hydrosulphurous acid is added to the dilute cupric sulphate solution, which is rapidly turned from blue to green. As the reaction proceeds, the solution changes to a dark brown, and a precipitate is obtained consisting of metallic copper and cuprous hydride.

The addition of sulphurous acid to cupric sulphate solution produces no precipitate.

Solutions of H_2SO_2, H_2SO_3, $HgCl_2$, $AgNO_3$, $CuSO_4$.

SULPHUR TRIOXIDE

PREPARATION

59. By the union of oxygen and sulphur dioxide in the presence of platinized asbestos. — (*a*) When a mixture of

sulphur dioxide and oxygen is passed over heated platinized asbestos, sulphur trioxide is formed.

A 500 cc. 3-necked Wolff bottle is half filled with concentrated sulphuric acid. A current of sulphur dioxide is conducted through a glass tube inserted in one of the necks of the Wolff bottle and dipping beneath the surface of the sulphuric acid. A glass tube, conducting oxygen, is thrust through another neck of the bottle and likewise dips beneath the surface of the acid. A short glass elbow is inserted in the cork in the third neck and is connected with a bulb-tube or combustion-tube containing a 3 or 4 cm. length of platinized asbestos (Fig. 69). The gases are passed with approximately the same degree of rapidity into the bottle and proceed unchanged through the platinized asbestos and bulb-tube into the air. On heating the platinized asbestos, however, dense white clouds of sulphur trioxide are formed.

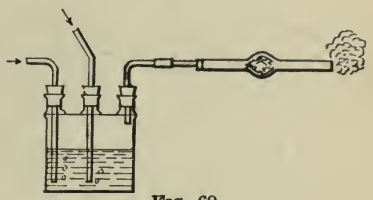

FIG. 69

$$2\ SO_2 + O_2 = 2\ SO_3.$$

500 cc. 3-necked Wolff bottle (Fig. 69); bulb-tube; SO_2 generator; O supply; Pt asbestos.

(*b*) Instead of passing a mixture of gaseous oxygen and sulphur dioxide over platinized asbestos, the simpler process of passing oxygen first through strong sulphur dioxide water, and then through concentrated sulphuric acid, and finally over platinized asbestos, may be used.

A current of oxygen is passed through a saturated solution of sulphur dioxide in water in a 200 cc. Erlenmeyer flask, which is gently warmed. The oxygen there mixes with the sulphur dioxide, and the two gases are conducted

through a gas washing-bottle containing concentrated sulphuric acid. The mixed gases are then conducted through a bulb-tube or combustion-tube containing platinized asbestos. On heating the platinized asbestos dense clouds of sulphur trioxide issue from the tube.

200 cc. Erlenmeyer flask ; bulb with platinized asbestos ; H_2SO_4 gas washing-bottle ; O supply ; saturated solution of SO_2.

60. From fuming sulphuric acid.— Nordhausen or fuming sulphuric acid, consisting, as it does, essentially of a solution of sulphur trioxide in sulphuric acid, readily liberates the trioxide on heating. The fuming acid is placed in a 500 cc. retort, whose neck is thrust into a 250 cc. flask immersed in a freezing-mixture of salt and ice. On gently heating the acid the sulphuric trioxide is given off and condenses in the receiver. Owing to the intensely corroding properties of the acid the retort must be very carefully heated to avoid danger of breakage. The danger is minimized if a sand-bath is used.

$$H_2S_2O_7 = H_2SO_4 + SO_3.$$

500 cc. retort ; 250 cc. flask ; sand-bath ; Nordhausen sulphuric acid.

61. From sulphuric acid and phosphorus pentoxide. — Phosphorus pentoxide abstracts water from sulphuric acid, setting free sulphuric anhydride.

A few drops of strong sulphuric acid are placed in a test-tube, and sufficient phosphorus pentoxide is added to make a thin paste. On gently heating the mixture a vigorous reaction takes place, clouds of sulphur trioxide being given off.

$$3\,H_2SO_4 + P_2O_5 = 2\,H_3PO_4 + 3\,SO_3.$$

62. By heating potassium disulphate. — Potassium disulphate, when heated in a test-tube, forms potassium sulphate and sulphur trioxide.

A bell-jar whose sides have been moistened with water is held over the test-tube containing potassium disulphate and the fumes allowed to condense in the bell-jar. On washing out the contents of the jar into a cylinder and adding barium chloride solution, a white precipitate of barium sulphate will be formed.

$$K_2S_2O_7 = K_2SO_4 + SO_3.$$

Bell-jar; $K_2S_2O_7$; $BaCl_2$ solution.

PROPERTIES

63. Action on water. — (*a*) A bottle containing sulphur trioxide fumes on removing the stopper. The fumes of Nordhausen or fuming sulphuric acid are due to the presence of large quantities of the trioxide.

The anhydride is very deliquescent, and, when solid, is best removed with a glass rod. Enough will cling to the rod to illustrate its properties.

The fundamental precaution, not to pour water into sulphuric acid, may be here repeated. The danger is infinitely greater if water is poured into a vessel containing sulphur trioxide. The results of such an operation are seen in the following experiments. The acid nature of a solution of sulphur trioxide in water may be shown by immersing a piece of blue litmus paper in it.

$$SO_3 + H_2O = H_2SO_4.$$

(*b*) The intensity of the reaction between sulphuric anhydride and water is so great that the addition of water to the anhydride becomes a very dangerous operation. On the small scale, the experiment may be made without danger, as follows: —

A small quantity of sulphur trioxide is placed in a platinum crucible, imbedded in a layer of sand, at the bottom of

a glass cylinder (Fig. 70). A glass funnel is inserted in the mouth of the cylinder, and its stem, which should be as long as possible, directed so as to allow water to drop into the crucible. On pouring a few drops of water into the funnel, they will fall upon the trioxide in the crucible, the union taking place with almost explosive violence.

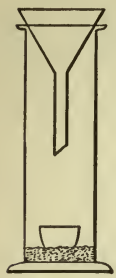

Fig. 70

Apparatus (Fig. 70); glass cylinder; Pt crucible; sand; funnel; SO_3.

(*c*) A tall cylinder is one-third filled with water and a few crystals, or 5 cc. of liquid sulphur trioxide are dropped into it. As each particle comes in contact with the water, there is a hissing and explosion. The column of air in the cylinder intensifies the sound.

Cylinder one-third filled with water; SO_3.

(*d*) A small dry test-tube is drawn out at the middle, so as to leave a hook of glass at the bottom of the upper part, to which a weight may be fastened (Fig. 71). One cubic centimeter of liquid sulphur trioxide, or its equivalent amount in crystals, is placed in the test-tube to which the weight has been fastened. It is then allowed to drop into a tall cylinder containing about a liter of water. The cylinder should not be more than three-fourths full. As the tube sinks beneath the surface of the water, the sulphur trioxide unites with the water with explosive violence.

Fig. 71

Gauntlets; drawn-out test-tube with weight; liter cylinder; SO_3.

64. Action of sulphur. — Sulphur dissolves in sulphur trioxide to form a blue liquid, the so-called sulphur sesquioxide.

On standing, the liquid decomposes, loses its blue color, and forms sulphur dioxide.

A small quantity of sulphur trioxide is placed in a dry test-tube, and a very small pinch of sulphur flowers added to it. The solution becomes blue, but by gently warming, the color disappears, and a paper moistened in potassium dichromate solution indicates the presence of sulphur dioxide.

$$SO_3 + S = S_2O_3. \quad [?]$$

Sulphur trioxide ; S flowers.

65. Action with phosphorus. — Phosphorus reduces sulphur trioxide, with the liberation of sulphur dioxide.

Two cubic centimeters of liquid sulphur trioxide are placed in a dry test-tube, and a 5 mm. piece of well-dried phosphorus is added. The phosphorus catches fire of itself and burns on the surface of the liquid, obtaining its oxygen from the sulphur trioxide. Large quantities of sulphur dioxide are evolved. The residue in the test-tube at the end of the reaction may be carefully immersed in a large cylinder of water, and it will be seen that the phosphorus has all been oxidized, as there will be little, if any, insoluble matter in the residue. Owing to the fumes evolved, it is better to conduct the experiment under the hood.

$$5\ SO_3 + 2\ P = P_2O_5 + 5\ SO_2.$$

SO_3 ; P.

66. Action with barium oxide. — Barium oxide combines with sulphur trioxide to form barium sulphate.

A small quantity of sulphur trioxide is placed in a dry test-tube, and powdered barium oxide carefully sifted into the tube. The union of the two compounds is accompanied by an evolution of heat and light.

$$BaO + SO_3 = BaSO_4.$$

Screens ; gauntlets ; barium oxide ; sulphur trioxide.

SULPHURIC ACID

FORMATION AND PREPARATION

67. By the oxidation of sulphur with nitric acid. — Sulphur, when heated with fuming nitric acid, is oxidized to sulphuric acid.

Two grams of sulphur flowers are heated to boiling in a 100 cc. Erlenmeyer flask with 10 cc. of fuming nitric acid. On diluting the mixture with water and pouring it upon a filter, the filtrate will be found to contain considerable free sulphuric acid, which will give a white precipitate with barium chloride.

S flowers; fuming HNO_3.

68. By the action of nitric acid on sulphur dioxide. — Sulphur dioxide, when conducted into concentrated nitric acid, becomes oxidized, forming sulphuric acid and setting free nitrogen peroxide. In case dilute nitric acid is used, nitric oxide rather than nitrogen peroxide is liberated.

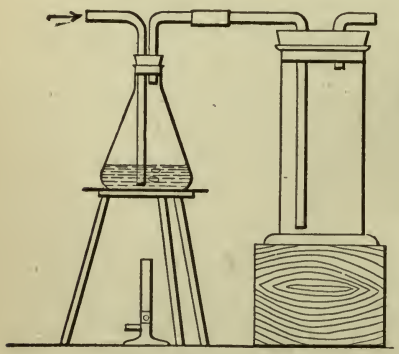

FIG. 72

A current of sulphur dioxide is conducted into a 300 cc. flask containing 75 cc. of concentrated nitric acid. The cork of the flask is provided with two holes, through one of which the sulphur dioxide is conducted, while the other allows for the escape of the nitrogen peroxide formed. An elbow in the second hole conducts the nitrogen peroxide to the bottom of an

empty glass cylinder fitted with a two-holed cork (Fig. 72). All connections should be made with the glass tubes as nearly touching each other as possible, since the nitrogen peroxide fumes attack rubber. On conducting sulphur dioxide into the nitric acid, the liquid warms up of itself and deep reddish-brown fumes of nitrogen peroxide escape and fill the glass cylinder. After a few moments the liquid in the flask may be diluted with water and tested for the presence of sulphuric acid by means of barium chloride.

If the concentrated nitric acid in the apparatus is replaced by an acid of the specific gravity of 1.15 and the experiment repeated, it will be found that the gas escaping into the glass cylinder, while somewhat colored, consists chiefly of nitric oxide, as can be seen by opening the mouth of the cylinder and allowing the gas to come in contact with the air. The formation of red fumes indicates the presence of nitric oxide. It will be found necessary to warm the dilute nitric acid somewhat in order to start the reaction. At the end of the experiment, the liquid may be tested as before for sulphuric acid.

$$SO_2 + 2\,HNO_3 = H_2SO_4 + 2\,NO_2.$$

$$3\,SO_2 + 2\,HNO_3 + 2\,H_2O = 3\,H_2SO_4 + 2\,NO.$$

Flask with 2-holed cork; glass cylinder; SO_2 generator; HNO_3 (sp. gr. 1.15); con. HNO_3.

69. Chamber crystals by the action of concentrated nitric acid on sulphur dioxide. — Nitric acid oxidizes sulphur dioxide, forming sulphuric acid.

A piece of asbestos paper fastened to a wire is dipped into concentrated nitric acid and lowered into a cylinder of sulphur dioxide. A marked reaction, filling the jar with red fumes, is observed. The walls of the cylinder soon become coated with chamber crystals.

Jar of SO_2; con. HNO_3.

70. By the oxidation of sulphur dioxide by means of nitric oxide in the presence of air and water vapor. — In the manufacture of sulphuric acid on a large scale the oxidation of sulphur dioxide is effected by means of nitric oxide in the presence of air. On the lecture-table the substitution of a gaseous for a liquid oxidizing agent may be successfully carried out by conducting the gaseous compounds, *i.e.* sulphur dioxide, nitric oxide, air, and water vapor, into a large flask, where the reaction takes place.

A 4 or 5 l. flask is provided with a four-holed cork, through three holes of which glass tubes are thrust about halfway to the bottom of the flask. In the fourth hole a short glass elbow is inserted, which connects with a flue (Fig. 73). The flask must be perfectly dry and is first filled with nitric oxide from a generator, consisting of a 300 cc. Erlenmeyer flask, fitted with a thistle-tube and a glass elbow. A layer of copper turnings is placed in the bottom of the generator and sufficient water added to cover them. Concentrated nitric acid is poured through the thistle-tube, and soon the reaction begins. The colorless nitric oxide combines with the oxygen of the air in the large flask, filling it with deep ruddy fumes of nitrogen peroxide. At this point the evolution of nitric oxide is stopped by pouring water into the nitric oxide generator. If it is desirable to start the action again, it is only necessary to add a little more concentrated nitric acid.

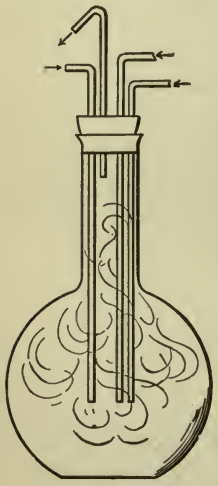

FIG. 73

The second tube in the cork of the large flask is connected with a sulphur dioxide generator, such as is de-

scribed in Ex. 41. The sulphur dioxide is first conducted through a gas washing-bottle containing a small amount of water to note the rapidity of its evolution. A vigorous stream of sulphur dioxide is then allowed to flow into the flask for two or three minutes. The third tube in the cork of the flask is connected with a 100 cc. Erlenmeyer flask containing 25 cc. of water and so provided with a cork and glass elbows that a stream of air may be blown through the water into the large flask. During the previous operations the water is brought almost to a boil. After the current of sulphur dioxide is stopped air is blown through the hot water, thereby carrying a small amount of moisture into the chamber. If care is taken not to admit too great a quantity of moisture, the sulphur dioxide and nitrogen peroxide will interact with the small amount of water, and the sides of the vessel will become covered with crystals, the so-called "chamber crystals."

The decomposition of the chamber crystals and the regeneration of the nitrogen peroxide are effected by passing into the flask considerable quantities of water vapor. The contents of the small flask are vigorously boiled, and the steam escapes into the large flask. The tube through which air is conducted, dipping under the surface of the water in the small flask, is sealed, and the steam is thus prevented from escaping into the air of the room. After the steam has entered the large flask for a few moments, the crystals are rapidly decomposed with effervescence, nitric oxide being liberated. By blowing air into the flask, its contents once more become red from the formation of nitrogen peroxide. If sulphur dioxide is now conducted into the flask, the red fumes will disappear, but the excess of moisture in the flask will probably prevent the reappearance of the chamber crystals.

In case the flask becomes too warm from the reaction, the

formation of chamber crystals may be somewhat accelerated by cooling the outside of the flask with a cloth wet with cold water. When the crystals are once started, they spread with considerable rapidity all over the surface of the flask.

If the formation of sulphuric acid only is desired, and the introduction of chamber crystals avoided, it is only necessary to conduct simultaneously into the large flask nitric oxide, sulphur dioxide, water vapor, and air. On adding water and pouring out the contents of the flask, they will be found to give a strong test for sulphuric acid with barium chloride.

$$SO_2 + H_2O + NO_2 = H_2SO_4 + NO.$$

$$2\,NO + O_2 = 2\,NO_2.$$

$$4\,SO_2 + 4\,NO_2 + O_2 + 2\,H_2O = 4\,SO_2\begin{cases} O \cdot NO. \\ OH. \end{cases}$$

4 or 5 l. flask; 4-holed cork and tubes; SO_2 generator; NO generator; steam generator,

71. From sulphur dioxide, fuming nitric acid, air, and water vapor. — The technical manufacture of sulphuric acid is considered of sufficient importance to demand a special and elaborate treatment in most text-books on chemistry. It is desirable, therefore, to present on the lecture-table a demonstration of the technical process for the manufacture of this acid, which shall not be too elaborate and yet shall be sufficiently detailed to show the essential steps. While the fundamental reactions are admirably illustrated in the apparatus described in the preceding experiment, a number of technical features of economic importance warrant illustrative consideration on the lecture-table, and accordingly the apparatus shown in Fig. 74 has been devised for this purpose.

The chamber consists of a 2 or 3 l. three-necked Wolff bottle, through the middle neck of which is inserted a two-holed rubber stopper carrying a dropping-funnel and a glass elbow. The other two necks are provided with one-holed rubber stoppers carrying glass tubes of the special form indicated in the figure. Two Liebig condenser jackets filled with

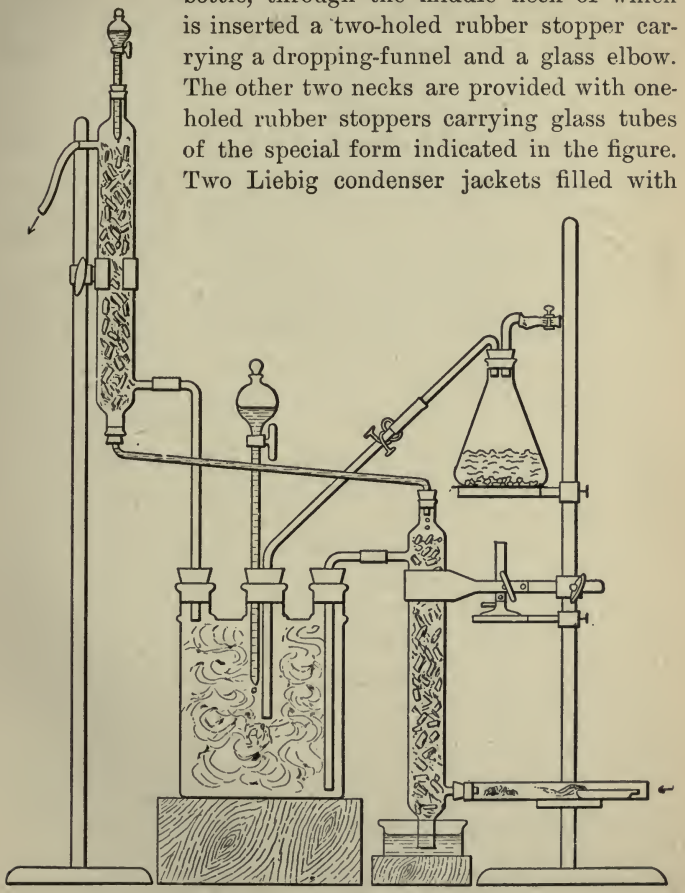

Fig. 74

broken bits of pumice-stone serve as the Gay-Lussac and Glover towers, respectively. The condenser jackets should

have the water-tubes on opposite sides if possible, as otherwise it will be necessary to bend glass tubes to make the proper connections. One of the condensers is clamped in an upright position, its lower end dipping into a small beaker one-third filled with concentrated sulphuric acid. A piece of combustion-tube 25 cm. long is fastened to the lower water-tube of the condenser by means of a rubber stopper, and a small plug of glass wool is thrust into the combustion-tube. A porcelain boat, filled with bits of roll sulphur of such a size as can conveniently pass into the combustion-tube, is inserted part way in the tube. In case the combustion-tube does not remain in a level position, it may be necessary to support it with a ring from the large retort stand.

The upper water-tube of the condenser is connected by a long glass elbow to one of the necks of the Wolff bottle. The second condenser jacket is clamped in a vertical position on the other side of the Wolff bottle, in such a manner that its lower end will be 3 or 4 cm. higher than the upper end of the other condenser. The two condensers are connected by a long, small-bored glass tube, thrust through the corks inserted in the end of the condensers. The glass tube should extend several millimeters below the cork in the top of the lower condenser, and should be just flush with the end of the cork thrust into the lower end of the upper condenser. As this cork is subject to the action of concentrated sulphuric acid, it is best to coat it with paraffin and thrust it, while still warm, into the condenser. Care should be taken in coating the cork that the paraffin does not seal the glass tube by running into it and solidifying. The lower water-tube of the upper condenser is connected with a long glass elbow to the third neck of the Wolff bottle. The upper water-tube is connected with a suction-pump, a gas washing-bottle containing water being inserted at some point between

the condenser tube and the pump. A one-holed rubber stopper, carrying a dropping-funnel filled with concentrated sulphuric acid, is inserted in the upper end of the upper condenser. Concentrated sulphuric acid is allowed to flow down over the pumice-stone in the upper condenser, which it thoroughly drenches. The acid collects in the bottom of the condenser and flows through the small tube into the lower condenser, trickling down over the pumice-stone, and finally is collected in the beaker.

The following arrangement is provided for sending water vapor through the glass elbow in the middle neck of the Wolff bottle. A 100 cc. Erlenmeyer flask, one-fourth filled with water, is fitted with a two-holed rubber stopper. A glass elbow, 7 mm. in diameter, is thrust through one hole in the cork, and a small glass elbow, 2 mm. in diameter, is thrust through the second hole. The large elbow carries a piece of rubber tubing and a pinch-cock. The small elbow is connected with the elbow leading into the middle neck of the Wolff bottle. The flask is supported on a stand and gently warmed with a Bunsen burner, the water being kept at or near boiling. The pinch-cock should be moved up on the glass elbow, thus allowing free escape for the water vapor through the large glass elbow and the rubber tube. Steam condenses in one end of the small glass elbow, and the drop clinging to the end of the tube prevents the escape of the steam into the Wolff bottle. When it is desired to introduce steam into the acid chamber, the flame is raised and the water in the flask brought to a vigorous boil. By closing the rubber tube on the large elbow with the pinch-cock, the steam forces its way through the small elbow into the acid chamber. On releasing the pinch-cock, the steam issues from the large elbow into the air of the room, as before. A few bits of pumice-stone or glass beads promote the regularity of ebullition.

The preparation of sulphuric acid by means of this apparatus is extremely simple. Ten to fifteen drops of fuming nitric acid are allowed to fall into the Wolff bottle, which should be perfectly dry. If the stem of the dropping-funnel has been previously filled with acid, the regulation of the dropping will be much simplified. The boat containing sulphur is then drawn halfway out of the tube, and the sulphur strongly ignited by heating with a lamp. When burning well, the suction is started and air drawn through the combustion-tube over the burning sulphur into the apparatus. After the current of air is started, it is necessary to maintain it at a rate just sufficient to draw the flame of the burning sulphur into the combustion-tube. In this manner a mixture of air and sulphur dioxide is carried into the chamber. After a few moments the walls of the flask will become covered with a deposit of chamber crystals. Water vapor is then admitted, and consequently the chamber crystals are decomposed with the liberation of nitrous fumes.

The formation of chamber crystals, being dependent on a certain proportion of water, nitric oxide, and sulphuric acid, is a matter of considerable difficulty, and cannot always be relied on. When the operation is carried out as described, however, the chamber crystals are, as a rule, readily obtained.

The interaction of the various products in the acid chamber is accompanied by a formation of fumes, showing that chemical reaction has taken place. On disconnecting the tubes and removing the Wolff bottle, it will be found that the contents diluted with water will give a strong test for sulphuric acid. When sulphur is burned in this manner a portion of it sublimes, but it is retained by the glass wool in the combustion-tube. The current of air entering the combustion-tube passes through the Glover tower into the chamber. Any nitrogen oxides carried away by the current of air are absorbed by the sulphuric acid in the Gay-Lussac

tower. The acid containing the nitrogen fumes flows down through the long glass tube into the upper end of the Glover tower, where, in trickling down over the pumice-stone, it comes in contact with the sulphur dioxide, which reacts with it, setting free the nitrogen oxides.

While the actual absorption of the nitrogen oxides in the Gay-Lussac tower, and their subsequent regeneration in the Glover tower, cannot be observed, the process is interesting as being a duplicate of that used in the commercial manufacture of this acid. The supply of sulphuric acid in the dropping-funnel in the top of the Gay-Lussac tower should occasionally be replenished, as the acid should continuously drop upon the pumice-stone.

Obviously, nitric oxide, instead of fuming nitric acid, may be conducted through the middle neck, in case the gaseous oxidizing agent is desired.

Apparatus (Fig. 74); 3-necked Wolff bottle; two Liebig condenser jackets filled with pumice-stone; dropping-funnel; porcelain boat; combustion-tube; glass wool; steam generator; suction-pump; S; fuming HNO_3.

PROPERTIES

72. Intense acidity. — The intense acid nature of sulphuric acid is shown by adding 1 drop of the concentrated acid to 2 l. of water in a large beaker. It will be found that the water is acid enough to redden a strip of blue litmus.

2 l. beaker; blue litmus paper.

73. Evolution of heat by mixing sulphuric acid with water. — (*a*) When sulphuric acid and water are mixed, there is considerable rise in temperature, sufficient to boil ether.

Two hundred cubic centimeters of water are placed in a 500 cc. flask, and 5 cc. of ether are added. On holding a

match at the mouth of the flask no flame will appear. On the addition of 100 cc. of concentrated sulphuric acid the temperature of the mixture is so increased that the ether boils vigorously, and the issuing vapor may be ignited at the mouth of the flask.

FIG. 75

After the flame has gone out, an ether thermometer, made by blowing an 8 or 10 mm. bulb on the end of a long piece of 3 mm. glass tubing, is half filled with ether, and the bulb thrust into the hot mixture of acid and water (Fig. 75). The ether boils vigorously, and on applying a flame burning ether vapor issues from the mouth of the tube

500 cc. flask; long tube with bulb; ether.

(*b*) The evolution of heat, on mixing these two liquids, is sufficient to give a temperature to the mixture of considerably over 100°. Sixty cubic centimeters of water and 120 cc. of concentrated sulphuric acid are simultaneously poured (to insure thorough mixing) into a 300 cc. beaker standing on wire gauze or asbestos paper. A thermometer inserted in the mixture will indicate a temperature of 115° to 120°.

A small quantity of alcohol in a thin-walled test-tube is immersed in the hot liquid. The alcohol boils, and the vapor may be ignited at the mouth of the tube.

Two cubic centimeters of water in a thin-walled test-tube may be quickly brought to a boil by immersing the tube in the hot solution.

300 cc. beaker; gauze; thin-walled test-tubes; 120 cc. con. H_2SO_4; alcohol.

74. Dehydrating action (drying of gases). — Sulphuric acid, owing to its intense hygroscopic properties, is of the

greatest importance in drying many gases. The interior surface of a bell-jar is moistened by holding it over a Bunsen or, better, a hydrogen flame. A small evaporating dish filled with lump pumice-stone, which has been drenched with sulphuric acid, is placed on a plate under the moistened bell-jar (Fig. 76). In a few minutes the walls of the jar will become clear and unclouded as the water vapor is rapidly absorbed by the sulphuric acid. The rapidity of the absorption is still more emphasized by preparing a second bell-jar as before and placing it over pumice-stone drenched with water. In the second case the bell-jar remains clouded with the condensed water vapor.

FIG. 76

H flame ; 2 bell-jars ; 2 evaporating dishes ; 2 plates ; pumice-stone.

75. Carbonization of sugar by sulphuric acid. — Sixty grams of lump sugar are added to 45 cc. of warm water in a 500 cc. beaker. The sugar will soon dissolve in the water, forming a thick syrup. The beaker is placed on a white plate and 60 cc. of concentrated sulphuric acid rapidly poured into it. The reaction is almost instantaneous, the solution blackening, and large quantities of steam being driven off. The residue is a porous, carbonaceous mass. The mixture will froth up and run over the edge of the beaker upon the plate.

500 cc. beaker ; lump sugar.

76. Action on paper. — Dilute sulphuric acid has no appreciable effect on paper, but if paper moistened with dilute acid is gently warmed, the water is vaporized, leaving the non-volatile sulphuric acid.

A dilute sulphuric acid is made by adding 10 cc. of con-

centrated sulphuric acid to 250 cc. of water. A letter is made on paper with this dilute acid, and the paper then allowed to dry in the air. When perfectly dry no change is noticeable. If the paper is held high over a Bunsen flame and simply warmed, the acid, concentrated by evaporation, will carbonize all portions of the paper with which it is in contact, and the character will appear in a charred outline.

SELENIUM

SELENIUM

1. Sublimation. — Selenium, when heated, sublimes without melting.

A small piece of selenium is heated in a dry test-tube. The element sublimes, and the upper part of the sublimate will consist of fine needles of selenium dioxide formed by the combustion of the vapor in the air of the tube.

2. Combustion in air. — Selenium, when heated in the air, burns with a blue flame, forming selenium dioxide.

A small piece of selenium is heated on a crucible cover and ignited. It burns with a characteristic blue flame, but it is necessary to keep the crucible cover heated during the combustion.

Crucible cover ; Se.

3. Solubility in strong sulphuric acid. — (*a*) Powdered selenium is covered in a test-tube with fuming sulphuric acid and strongly heated. The selenium dissolves with the liberation of sulphur dioxide. The sulphuric acid containing selenious acid becomes green. The green solution is carefully poured into a 300 cc. cylinder half filled with cold water. Selenium separates out of the solution in the form of a fine, dark red powder.

Se ; fuming H_2SO_4 ; $K_2Cr_2O_7$ solution.

(*b*) Selenium, when heated with fuming sulphuric acid, dissolves with a green color. If the solution is then diluted with concentrated sulphuric acid and heated to boiling for a few moments, it will finally be decolorized as the selenium is oxidized by the sulphur trioxide. On pouring the decolorized solution into water no precipitate of selenium will occur.

$$2\ SO_3 + Se = 2\ SO_2 + SeO_2.$$

Fuming H_2SO_4; Se.

SELENIUM DIOXIDE

4. Preparation. — Selenium burns in a current of oxygen to form selenium dioxide. A few lumps of selenium are placed in a bulb-tube, which is fitted to the mouth of a dry filter-flask, the side tube of which is connected with a flue (Fig. 77). A current of oxygen is passed through the bulb-tube and the selenium heated to ignition. The product condenses on the arm of the tube and in the receiver as a white powder.

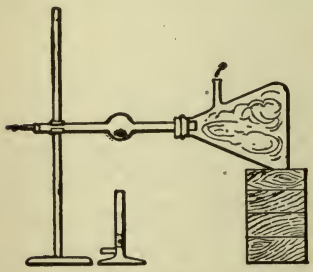

FIG. 77

$$Se + O_2 = SeO_2.$$

Apparatus (Fig. 77); bulb-tube; filter flask; O supply; Se.

5. Green color of the vapor. — A small quantity of selenium dioxide is heated in a test-tube. The greenish vapor is best seen by holding the tube in front of a white background.

SELENIOUS ACID

6. Oxidation of selenium with nitric acid. — When powdered selenium is heated in a test-tube with concentrated nitric acid, it rapidly dissolves, with the liberation of nitrous

fumes, forming selenious acid. After neutralization with ammonia the solution gives any of the reactions for selenious acid.

7. By dissolving selenium dioxide in water. — Selenium dioxide dissolves readily in water, forming selenious acid. The aqueous solution may be used in the following experiments.

8. Action with hydrogen sulphide. — Hydrogen sulphide solution, when added to a solution of selenious acid, produces a bright yellow precipitate of selenium sulphide.

9. Reduction by sulphurous acid. — Sulphurous acid reduces selenious acid to selenium. Sulphur dioxide, or a solution of sulphur dioxide in water, is added to a dilute solution of selenious acid. In the cold, or more rapidly by warming, a brick-red precipitate of finely divided selenium is obtained.

$$H_2SeO_3 + 2\ SO_2 + H_2O = 2\ H_2SO_4 + Se.$$

H_2SO_3; H_2SeO_3.

SELENIC ACID

10. Preparation. — Chlorine water oxidizes selenium dioxide to selenic acid.

Selenium dioxide is covered with chlorine water and gently warmed; sufficient chlorine water should be added to complete the oxidation, and, on boiling off the excess of chlorine, selenic acid is obtained.

$$SeO_2 + Cl_2 + 2\ H_2O = H_2SeO_4 + 2\ HCl.$$

SeO_2; chlorine water.

NITROGEN

NITROGEN

PREPARATION

1. By the decomposition of ammonium chloride and sodium nitrite. — The decomposition of ammonium nitrite results in the formation of nitrogen and water according to equation (1) below. The instability of ammonium nitrite precludes its use in the pure state for this reaction, hence it is prepared and instantly decomposed by using ammonium chloride and sodium nitrite, whose interaction produces sodium chloride and ammonium nitrite, equation (2).

A mixture of 10 g. of ammonium chloride and 15 g. of sodium nitrite is placed in a 200 cc. Erlenmeyer flask fitted with a thistle-tube and a delivery-tube. Thirty cubic centimeters of water are then added and the flask gently heated. The nitrogen is liberated considerably below the boiling point of water, and it is advisable not to overheat the mixture, as frothing is likely to occur. In case the frothing is too strong, the introduction of a few cubic centimeters of water through the thistle-tube will immediately check it. Nitrogen may be collected at the pneumatic trough in cylinders.

(1) $NH_4NO_2 = N_2 + 2\ H_2O$.

(2) $NaNO_2 + NH_4Cl = NaCl + NH_4NO_2$.

200 cc. Erlenmeyer flask; thistle-tube and delivery-tube; NH_4Cl; $NaNO_2$.

2. By the ignition of a mixture of ammonium chloride and potassium dichromate. — The decomposition of ammonium dichromate yielding nitrogen, water, and chromic oxide is described in Ex. 6, p. 361. As ammonium dichromate is rather deliquescent and not common in the laboratory, the same results may be obtained by using a mixture of ammonium chloride and potassium dichromate.

Eight grams of powdered ammonium chloride and 25 g. of powdered potassium dichromate are intimately mixed and placed in a 100 cc. Jena glass Erlenmeyer flask. The flask is fitted with a very wide delivery-tube, preferably 1 cm. internal diameter, which leads to a pneumatic trough (Fig. 128, p. 319). On heating the mixture nitrogen is evolved and some of the ammonium chloride is sublimed, hence the necessity of a wide delivery-tube. The Jena glass flask will stand a very high heat without danger.

$$2\ NH_4Cl + K_2Cr_2O_7 = 2\ KCl + Cr_2O_3 + 4\ H_2O + N_2.$$

100 cc. Jena glass Erlenmeyer flask ; wide delivery-tube ; $K_2Cr_2O_7$; NH_4Cl.

3. From potassium nitrate and iron powder. — Iron powder combines with the oxygen of potassium nitrate, liberating nitrogen.

A mixture of 10 g. of iron filings and .5 g. of powdered potassium nitrate is heated in a hard-glass test-tube fitted with a delivery-tube leading to the pneumatic trough. When the mixture is heated, a gas is rapidly evolved which on testing is shown to be nitrogen.

Hard-glass test-tube ; cork and delivery-tube ; KNO_3 ; Fe powder.

4. By abstraction of oxygen from air by means of phosphorus. — The commonest source of nitrogen is air, which consists essentially of nitrogen mixed with oxygen. To

remove the oxygen some material, such as phosphorus, which forms a solid or non-gaseous oxide, is selected.

A flat cork, some 2.5 or 3 cm. in diameter and a centimeter thick, has the ring of a porcelain crucible lid pressed into a slit in one side, and on the other side a small weight of lead or iron is fastened to give the apparatus when floated on water a greater stability. The float is placed on the surface of the water in a pneumatic trough and a 5 mm. piece of well-dried phosphorus laid on the crucible lid. A tubulated bell-jar, with a rather wide mouth to permit of the insertion of a candle on a wire, is tightly corked and placed immediately above the float (Fig. 78). The phosphorus is ignited by touching with a hot iron wire, and the bell-jar immediately lowered until its mouth is sealed with water. The phosphorus burns rapidly, and the heat generated by the combustion causes the gas in the bell-jar to expand and bubble out to a certain extent under the mouth of the bell-jar. As soon as the phosphorus goes out, however, the water will rise in the bell-jar, indicating a marked contraction in volume. On lowering the bell-jar until the level of the liquids inside and outside is the same, the cork may be removed and a burning candle on the end of a wire lowered into the residual gas. It will be immediately extinguished.

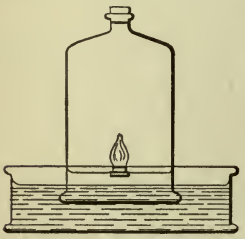

FIG. 78

Large cork ; crucible cover ; tubulated bell-jar ; candle on wire ; P.

5. By the removal of oxygen from the air by burning hydrogen. — When hydrogen is burned in air water is formed, the greater portion of which is immediately condensed. Hydrogen from a Kipp generator is passed through a glass tube (Fig. 79) so bent as to rise under a tubulated bell-jar and

have its tip at least 5 cm. above the level of the water. The glass tube is provided with a platinum tip consisting of a small piece of foil rolled around a glass rod and inserted in the end of the glass tube. After lighting the hydrogen flame, which should not be too high, the tubulated bell-jar, securely corked at the tubulature, is lowered over the burning jet. The oxygen is rapidly burned to form water, which deposits on the sides of the jar as a mist. As it is somewhat difficult to observe the hydrogen flame, it is best to moisten the tip of the platinum with a little sodium chloride solution to impart a color to the flame. The instant the flame is extinguished the supply of hydrogen should be cut off and the burner removed, as otherwise the admission of unburned hydrogen will contaminate the nitrogen. The level of the water on the inside of the bell-jar will have risen considerably and, as in the preceding experiment, the bell-jar should be lowered until the inner and outer levels are the same before removing the stopper. The tubulature should be large enough to permit the introduction of a burning candle in testing the gas. It is of the utmost importance that the hydrogen flame should be watched and the supply of hydrogen cut off the moment the flame is extinguished. This separation of oxygen and nitrogen is extremely simple and not difficult of comprehension by elementary students, as oxygen, hydrogen, nitrogen, and water are familiar substances.

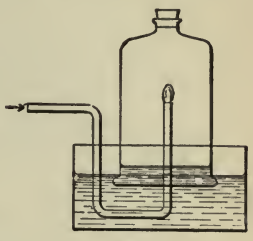

FIG. 79

Tubulated bell-jar ; jet with Pt tip ; H supply ; NaCl solution.

6. By the combustion of air and ammonia on copper.—When large quantities of nitrogen are desired, by far the most sat-

isfactory method for obtaining it is that in which a mixture of air and ammonia gas is passed over a hot copper coil, which is alternately oxidized by the air and reduced by the ammonia, forming water and nitrogen. The air is obtained from an ordinary water-blast, and the ammonia vapor from the strongest aqua ammonia, which is placed in a gas washing-bottle (Fig. 80). The air is allowed to bubble through the liquid and become saturated with the ammonia gas. The gaseous mixture is then passed into a 30 cm. length of combustion tubing containing a coil of copper wire and fitted with a delivery-tube at the other end leading to a pneumatic trough containing acidulated water. The copper coil is heated by means of a strong Bunsen burner at the end nearest the entrance of the gases. The copper will become oxidized by the oxygen of the air, and the copper oxide thus formed be immediately reduced by the ammonia gas, forming water and nitrogen. The reduced copper is then immediately oxidized and reduced, the process being a continuous one. The air, deprived of its oxygen by the copper, passes on as nitrogen into the pneumatic trough. To this nitrogen is, of course, added the nitrogen liberated from the ammonia. As the reaction proceeds, the copper coil becomes intensely hot and the external heat may be withdrawn. There should always be an excess of ammonia gas in the air current, and consequently all of the spiral but the front end should be in the reduced state. As soon as it begins to oxidize for any distance it is an indication that the supply of ammonia is becoming exhausted. By

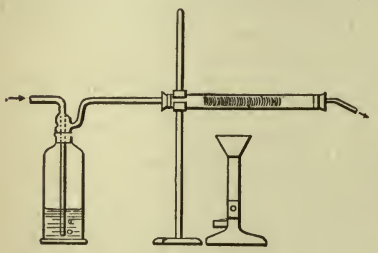

FIG. 80

observing the color of the spiral, the operation can be well regulated. The excess of ammonia vapor carried along to the pneumatic trough will be dissolved in the water, and unless large quantities of nitrogen are to be prepared and the water becomes saturated with the ammonia, no precaution is necessary. It may, however, be necessary after a while to have free sulphuric acid present to neutralize the ammonia as it is dissolved. When very strong ammonia water is used in the gas washing-bottle, it is found that a good strong air-blast is necessary.

$$2\ NH_3 + 3\ CuO = 3\ H_2O + 3\ Cu + N_2.$$

Air-blast; gas washing-bottle; combustion-tube; strongest NH_4OH; Cu coil.

7. Oxidation of nitrogen by burning magnesium in air. — The heat of burning magnesium is sufficient to cause a union of nitrogen and oxygen in small quantities.

An 8 cm. piece of magnesium ribbon is tied to a stout iron wire and after ignition quickly lowered into a clean, dry 500 cc. cylinder having a layer of sand on the bottom. The nitrogen and the oxygen of the air are caused to combine, and a strong test for nitrous acid is obtained with iodo-starch paper lowered into the cylinder. The layer of sand prevents the cylinder from cracking if bits of burning magnesium fall to the bottom.

500 cc. cylinder; Mg ribbon; KI-starch paper.

8. Formation of oxides of nitrogen by the combustion of hydrogen in oxygen. — The union of nitrogen and oxygen may be brought about by the intense heat of the hydrogen flame burning in oxygen mixed with a small quantity of air. A clean, dry Erlenmeyer flask of 1 or 2 l. capacity is filled with oxygen by displacement and a burning jet of hydrogen lowered into the flask. On account of the heat generated it

is necessary to have a platinum tip on the glass tube as is recommended in Ex. 5. The mouth of the flask being open, a certain amount of air can enter, and a portion of its nitrogen will combine with the oxygen to form nitrous acid. After allowing the hydrogen to burn for a few minutes the jet is withdrawn and moistened iodo-starch paper is held in the flask. It is immediately colored blue, indicating the presence of nitrous acid.

Jet (Fig. 41, p. 85) ; H generator; jar of O ; KI-starch paper.

ATMOSPHERIC AIR

9. Determination of oxygen in air by potassium pyrogallate. — One hundred cubic centimeters of air are introduced into the eudiometer (Fig. 11, p. 26), and potassium pyrogallate solution (Ex. 21, p. 26) allowed to flow slowly down through the gas. The absorption will have ceased when the liquid stops rising in the tube. Before reading off the volume of the remaining gas the reagent should be washed out of the tube by allowing successive portions of water to flow through the stop-cock. It will be found that the volume has diminished 20 cc., or one-fifth.

The method is capable of yielding very accurate analyses if the eudiometer is lowered in taking each reading till the levels of the inner and outer liquids are the same. In exact measurements, however, fluctuations in the temperature of the gas must be avoided.

Eudiometer, Fig. 11, p. 26 ; potassium pyrogallate solution.

10. Quantitative determination of nitrogen in air. — While the combination of burning phosphorus and oxygen attended by light and heat is extremely rapid, the elements do, nevertheless, unite at ordinary temperatures.

One hundred cubic centimeters of air are introduced into a eudiometer tube. A stick of phosphorus 3 or 4 cm. long is carefully cleaned under water and fastened to a piece of copper wire. The phosphorus is then thrust under the mouth of the eudiometer tube, which remains under water and is pushed a considerable distance up the tube, into the air. The copper wire is then bent so as to rest on the bottom of the dish and support the phosphorus in the air (Fig. 81). The level of the water in the tube will, of course, be depressed by as much as the volume of the copper and the phosphorus introduced. Hence it is important that the eudiometer be of sufficient size to allow of this expansion over the 100 cc. After the whole apparatus has stood over night it will be found that a diminution in volume has taken place. By withdrawing the phosphorus and reading off the volume of the residual gas it will be found to be approximately 80 cc., *i.e.*, four-fifths of the original volume.

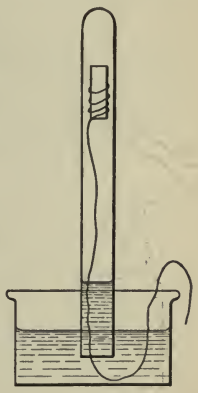

FIG. 81

In case a graduated tube is not at hand a linear measurement of the diminution may be made.

Eudiometer tube 2 cm. in diameter ; meter stick ; P ; Cu wire.

11. Quantitative absorption of oxygen from air by metallic copper. — By measuring the quantity of air passed over a heated copper coil and the amount of gas collected at the pneumatic trough, the proportion of oxygen and nitrogen present in the air may be quantitatively determined.

A stoppered cylinder is fitted with a two-holed rubber stopper carrying one tube leading to the bottom of the cylinder and a glass elbow directly connected with the combustion-tube

containing the copper coil. Connection is then made with a faucet, so that by opening the valve, water may be allowed to flow into the glass cylinder, expelling the air at the top through the elbow into the combustion-tube. After making all the connections the combustion-tube containing the copper is brought to red heat, the expanded air being allowed to escape at the pneumatic trough (Fig. 82). When a constant temperature has been reached, noted by the absence of air bubbles from the delivery-tube, an inverted graduated stoppered liter cylinder filled with water is placed over the

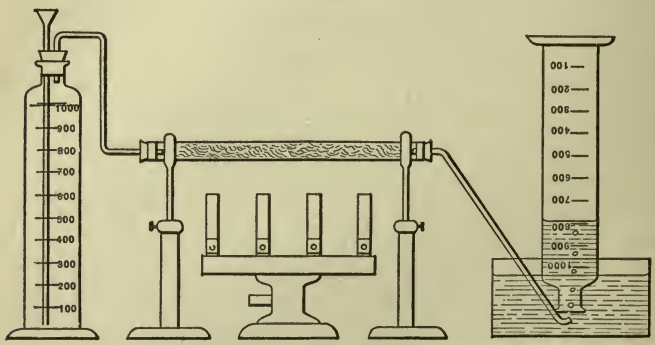

Fig. 82

delivery-tube and water allowed to flow slowly into the first graduate. The rate must not be too rapid, as otherwise the absorption of oxygen would not be complete. Water is allowed to flow into the first cylinder until it has reached the 1000 cc. graduation. The volume of gas collected at the pneumatic trough will be found to be about 800 cc. More exact measurement may be made by lowering the graduate into a deep vessel until the inner and outer levels of the water are the same. It is thus seen that from 1000 cc. of air, approximately 800 cc. of nitrogen remain after the

absorption of 200 cc. of oxygen. Both cylinders should be protected from any undue heating by asbestos screens placed between them and the burner.

Two 1000 cc. graduated stoppered cylinders; combustion-tube; Cu coil.

12. Quantitative combustion of phosphorus in a confined volume of air. — The regularity of the combustion of red phosphorus in air makes this form of phosphorus better adapted for the experiment in which oxygen is burned out of a confined volume of air.

A crucible lid containing red phosphorus is arranged as in Ex. 4, and a tubulated bell-jar placed over the float. A mark on the bell-jar is made about 2 cm. from the mouth and the remaining volume divided into fifths. The red phosphorus in the crucible lid is provided with a small piece of touch-paper, which is ignited. The bell-jar with the cork removed is lowered into water to the point marked 2 cm. above the mouth. The cork is then inserted, and when the phosphorus itself begins to burn the water in the bell-jar rises as the oxygen is consumed. The contraction of the gas (one-fifth of the original volume) is very accurately noted by means of this apparatus. The pneumatic trough should be deep enough to lower the jar at the end of the experiment till the inner and outer levels are the same.

Tubulated bell-jar (graduated in fifths); crucible lid on cork; red P; touch-paper.

AMMONIA

FORMATION AND PREPARATION

13. By the ignition of organic substances. — (*a*) A small quantity of gelatine or glue heated in a test-tube yields ammonia.

Gelatine or dry glue.

(*b*) The presence of ammonia may be established in the products of the dry distillation of coal (Ex. 62, p. 325) by inserting a piece of moistened red litmus paper in the filter-flask of the apparatus (Fig. 130, p. 325).

14. By heating organic nitrogenous matter with soda-lime. — While gelatine and similar compounds give nitrogen directly on ignition, many organic substances containing nitrogen — uric acid, for example — do not yield ammonia by simple heating.

A small quantity of uric acid heated in a test-tube gives no test for ammonia.

When such compounds are intimately mixed with dry soda-lime and ignited, their nitrogen is converted into ammonia. If uric acid is heated with an equal volume of fused dry soda-lime in a hard-glass test-tube, ammonia is evolved.

Uric acid ; dry soda lime.

15. By heating potassium nitrate, potassium hydroxide, and iron powder. — When iron powder is heated in the presence of potassium hydroxide, hydrogen is liberated in a manner similar to that shown in Ex. 5, p. 42. As is seen, however, in Ex. 3, the ignition of iron and potassium nitrate yields nitrogen. Heating equal volumes .5 g. each of potassium hydroxide and potassium nitrate with 20 g. of iron fillings in a hard-glass test-tube gives a copious evolution of ammonia.

Fe powder ; KNO_3 ; KOH.

16. From hydrogen and nitric oxide. — If a mixture of hydrogen and nitric oxide is passed over heated platinized asbestos 5 volumes of the hydrogen combine with 2 volumes of the nitric oxide and ammonia is formed.

Hydrogen from a Kipp generator is passed through a glass

tube thrust through a three-holed cork in the neck of a small bottle containing a 2 cm. layer of sulphuric acid. A stream of nitric oxide (Ex. 49, p. 211) passes through a second glass tube into the bottle. Both tubes dip beneath the surface of the sulphuric acid that their rate of bubbling may be noticed. Hydrogen is conducted through the whole apparatus to drive out all air and then the nitric oxide generator started. The current of hydrogen should be three times as fast as the current of nitric oxide. The mixed gases are then conducted through a piece of combustion-tubing or a bulb-tube containing platinized asbestos (Fig. 83). Until the asbestos is heated no ammonia is present in the issuing gases, which redden on exposure to the air; but on heating the asbestos a strong test for ammonia is immediately obtained and no red fumes are formed.

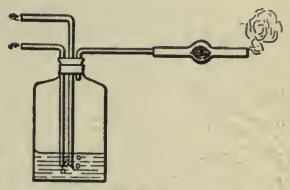

FIG. 83

$$2\,NO + 5\,H_2 = 2\,NH_3 + 2\,H_2O.$$

H generator; NO generator; wash-bottle, with 3-holed cork; bulb-tube; platinized asbestos.

17. From ammonium hydroxide and potassium hydroxide. Strong ammonium hydroxide is allowed to drop from a separating-funnel upon solid potassium hydroxide, preferably in the stick form, in a 500 cc. Erlenmeyer flask (Fig. 3, p. 11). The dropping-funnel is placed in a two-holed rubber stopper, and a glass elbow conducts away the gaseous ammonia liberated. In the process of the reaction the contents of the flask become very cold from the volatilization of ammonia, and consequently the gas is quite dry. However, in all experiments where a perfectly dry gas is required it should be first conducted through a U-tube containing dry quick-

lime or soda-lime. This method is by far the most convenient and available one for obtaining varying quantities of ammonia on the lecture table.

Apparatus, Fig. 3, p. 11; 500 cc. flask; dropping-funnel; stick KOH; con. NH_4OH.

18. By heating ammonium hydroxide. — One of the most convenient sources of gaseous ammonia is the strongest aqua ammonia of commerce. The simple application of heat suffices to drive off the ammonia which, when dried, is ready for use. The aqueous ammonia is placed in a flask fitted with a thistle-tube and a glass elbow. On gently warming, ammonia is driven off and passes through a gas washing-bottle containing a small quantity of strongest ammonia water, and then through a U-tube containing quicklime or fused soda-lime to dry the gas, which may be collected over mercury. The gas washing-bottle with the strong ammonia water is used to show by the bubbling the rate at which the gas is given off. Calcium chloride cannot be used to dry ammonia, as it forms a compound with the gas, and hence quicklime or soda-lime is recommended. The gas may be collected over mercury or by displacement.

Flask with thistle-tube and elbow; gas washing-bottle; U-tube with soda-lime; strongest NH_4OH.

19. From ammonium chloride and slaked lime.— Powdered ammonium chloride and slaked lime in equal quantities (about 40 g. of each) are placed in a 300 cc. Jena glass Erlenmeyer flask fitted with a safety-tube (such as is shown in Fig. 85, p. 196) and a glass elbow. A small quantity of concentrated ammonium hydroxide or mercury is placed in the bend of the safety-tube. On the application of gentle heat ammonia is rapidly evolved. The gas may be dried by conducting it through a U-tube containing quicklime or

fused soda-lime. Owing to its low specific gravity, ammonia can be readily collected by displacement of air according to the method of collecting hydrogen. Almost invariably this method of collecting the gas may be used.

$$2\ NH_4Cl + Ca(OH)_2 = CaCl_2 + 2\ H_2O + 2\ NH_3.$$

300 cc. Jena glass Erlenmeyer flask ; safety-tube ; soda-lime drying-tube ; NH_4Cl ; $Ca(OH)_2$.

PROPERTIES

20. Specific gravity. — Ammonia is considerably lighter than air, resembling hydrogen in this respect.

A jar of ammonia may be opened under the mouth of an inverted beaker suspended on the end of a balance which has been brought into equilibrium. On allowing the ammonia to rise into the beaker and expel the air the equilibrium will be disturbed.

Lecture-balance ; inverted beaker ; jar of NH_3.

21. Alkaline nature and tests.—(*a*) Ammonia gas imparts the color characteristic of alkalies to papers saturated with solutions of litmus, cochineal, turmeric, or phenol-phthalein. The papers may be held in a jar of ammonia or at the mouth of a bottle containing strong ammonium hydroxide.

Ammonia, however, is a volatile, as distinguished from a fixed, alkali, and if the papers colored by ammonia are allowed to remain in the air, the original colors, or in case of phenol-phthalein absence of color, will return.

Litmus, turmeric, cochineal, phenol-phthalein papers, or solutions of these indicators ; strong NH_4OH ; dry red litmus paper.

(*b*) **With gaseous hydrogen chloride.** — If a rod moistened with concentrated hydrochloric acid is held at the mouth of a test-tube from which ammonia is escaping, white fumes of ammonium chloride will be formed.

(*c*) **Action on mercurous nitrate.** — A piece of filter-paper dipped in mercurous nitrate solution is instantly turned black in the presence of ammonia.

$Hg_2(NO_3)_2$ solution.

22. Solubility in water. — A tall glass cylinder is filled with ammonia by displacement, covered with a metal disk, and opened under water by slowly sliding the disk to one side. The water rushes up into the cylinder with almost explosive violence. On account of this rapid solubility of the gas in water the use of a metal, rather than a glass, cover is recommended, as the latter might be broken by the rapid inrush of water.

Cylinder of NH_3; metal disk.

23. Solubility in water producing a fountain. — A 2 l. round-bottomed flask is filled with ammonia by displacement, the gas entering through a long tube pushed through a two-holed rubber stopper leading to the bottom of the flask, which is supported in an inverted position. When completely filled with ammonia, the cork and the tube are rapidly withdrawn and another two-holed rubber stopper, with fittings described beyond, is rapidly inserted in the neck of the flask. Through one of the holes in the cork a long glass tube, whose end has been drawn out to a 2 mm. opening, is thrust until the jet is about in the centre of the flask (Fig. 84). The other end of the tube, which must extend some 25 or 30 cm. beyond the cork, is plugged with a small piece of rubber tubing and a bit of glass rod, and dips into a crystallizing dish filled with a slightly acid solution of litmus. Through the other hole of the cork is thrust an ordinary medicine dropper which has been filled with water to within a few millimeters of the end of the jet. It is important to have the rubber bulb as well as the glass portion of the dropper

filled with water. The whole apparatus is firmly supported on the ring of a retort stand. On removing the plug from the end of the tube dipping under water no action takes place, as there is a long layer of air in the tube between the ammonia and water. On pinching the bulb of the dropper a few cubic centimeters of water are suddenly introduced into the flask, and absorption is instantaneous, the vacuum produced being sufficient to cause the water to ascend the long tube and play out of the jet as a fountain. The alkalinity of the water is strikingly shown by the change in color of the litmus solution.

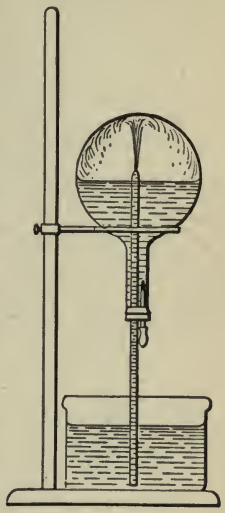

FIG. 84

In case the flask has been completely filled with gaseous ammonia, water will rush in till the flask is full. Provision must be made for the addition of water to the crystallizing dish as fast as it is withdrawn. The support must be strong enough to hold up the flask when filled with 2 l. of water.

2 l. flask (dry); 2-holed cork and jet; medicine dropper; NH_3 generator; soda-lime drying-tube; litmus solution.

24. Collection over mercury and absorption by water. — Gaseous ammonia, owing to its great solubility in water, can be collected only over mercury. Dried gaseous ammonia is collected in a thick-walled test-tube over mercury, care being taken not to have the delivery-tube dip too deeply into the mercury, thereby increasing the pressure to be overcome. The great solubility of ammonia is seen when a few drops of water are allowed to enter the tube through a

hooked glass tube and to rise through the mercury and come in contact with the gaseous ammonia. The absorption is very rapid, the mercury rising in the tube to take the place of the absorbed gas.

A piece of ice may be used instead of water.

Mercury trough ; NH_3 supply ; ice.

25. Preparation of ammonium hydroxide (ammonia water). — Ammonium hydroxide is prepared by saturating water with gaseous ammonia.

Ammonia gas is generated as in Ex. 19, and conducted directly from the flask without the introduction of a drying-tube into an empty gas washing-bottle which serves to collect any solid particles mechanically carried over.[1]

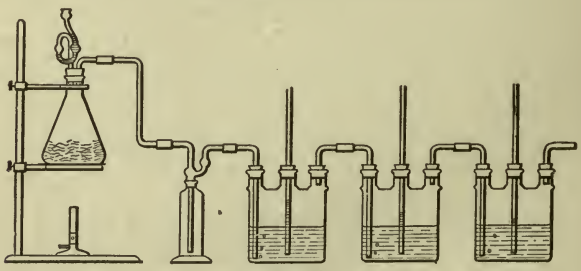

FIG. 85

The gas is then conducted through a series of three 3-necked Wolff bottles of 300–400 cc. capacity, each half filled with distilled water (Fig. 85). Each Wolff bottle is fitted with an elbow reaching to the bottom of the bottle, a vertical safety-tube dipping just beneath the surface of the water, and an elbow extending just below the cork. As soon as all the air is driven out of the generating flask, the bubbles

[1] When a large quantity of ammonia is desired, 100 g. each of ammonium chloride and calcium hydroxide should be heated in a 500 cc. flask.

rising through the water in the first of the series of Wolff bottles grow smaller, and finally all the gas is absorbed. As soon as the water in this bottle becomes nearly saturated, the gas rises through the solution unabsorbed and passes through the elbow into the second bottle, etc.

As the ammonium hydroxide is lighter than water, it is important that the glass elbows extend to the bottom of each bottle, as otherwise the upper surface of the liquid would become saturated, while the lower strata would be but slightly alkaline.

$$NH_3 + H_2O = NH_4OH.$$

Apparatus (Fig. 85) ; 500 cc. Jena glass Erlenmeyer flask ; three 3-necked Wolff bottles ; NH_4Cl ; $Ca(OH)_2$.

26. Absorption by charcoal. — Freshly ignited charcoal absorbs large quantities of ammonia.

Ammonia collected over mercury as in Ex. 24, is rapidly absorbed by a centimeter piece of heated charcoal immediately introduced under the mercury so as to come in contact with the gas. As the gas is absorbed, the mercury rises rapidly to take its place.

Tube filled with NH_3 over mercury ; charcoal.

27. Absorption by fused calcium chloride. — Fused calcium chloride absorbs ammonia, forming a definite chemical compound. For this reason calcium chloride, so often used as a dryer for gases, cannot be used for drying ammonia. Ignited soda-lime or quicklime is usually used for this purpose.

A small piece of fused calcium chloride is slipped under a tube filled with gaseous ammonia over mercury (Ex. 24). Immediately the absorption begins, and the calcium chloride melts.

Tube filled with NH_3 over mercury ; fused $CaCl_2$.

28. Absorption by silver chloride. — Silver chloride, as well as calcium chloride, absorbs ammonia, forming a definite compound. A few fragments of precipitated silver chloride (residues from chlorine determinations) are slipped under a test-tube of ammonia collected over mercury. The ammonia is rapidly absorbed, mercury rising to take its place.

On heating the silver compound ammonia is again expelled.

Tube filled with NH_3 over mercury ; AgCl.

29. Combustion in air. — (*a*) Ammonia, owing to its large percentage of hydrogen, is somewhat combustible in air, though only noticeably so under certain conditions.

The flame of a Bunsen burner is turned very low, and gaseous ammonia is conducted through a drawn-out glass jet bent at the end so as to deliver the gas into the centre of the burner. The glass jet is introduced upward into one of the air-holes. The ammonia will burn with a characteristic yellowish flame.

A very interesting study of the structure of the flame may be made by allowing a gentle stream of ammonia to enter the air-holes in the bottom of a Bunsen burner. The burner should give a good flame and be turned on full. The ammonia will impart a yellowish cast to the whole flame and the outlines of the several cones will be distinctly seen.

When a Bunsen burner is held at the mouth of a tube at which ammonia gas is escaping, the flame is colored yellow, and on close inspection it will be observed that particularly when the end of the tube becomes hot there is a distinct combustion of the ammonia itself.

NH_3 supply.

(*b*) Ammonia gas issuing from a glass jet will not continue to burn in air, as the temperature of its own flame is not sufficiently high to dissociate the ammonia gas. The necessary heat is easily furnished by passing the gas through the ring of flame at the mouth of a central draft burner.

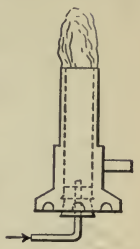

Fig. 86

The flame of an Erlenmeyer burner is turned as low as possible and yet leave a small ring of blue flame around the central draft tube (Fig. 86). In the base of this tube a cork carrying a glass elbow is inserted. Ammonia gas is led through the glass elbow and burns at the top with a yellowish flame.

Erlenmeyer burner ; NH_3 supply.

30. Combustion in oxygen. — A gentle stream of ammonia, generated by warming 20 cc. of the concentrated liquid in a 50 cc. Erlenmeyer flask, is made to enter an open tube filled with oxygen. The glass tube conducting the ammonia is passed through a two-holed rubber stopper fitted into the end of a short piece of wide glass tubing (Fig. 87). A gentle stream of oxygen is allowed to enter through a glass elbow and maintain an atmosphere of oxygen about the open tube leading from the ammonia flask. The ammonia burns with a distinctly luminous flame.

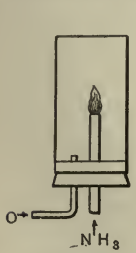

Fig. 87

Apparatus (Fig. 87) ; con. NH_4OH ; O supply.

31. Combustion of oxygen in ammonia. — A 150 cc. beaker is half filled with the strongest ammonium hydroxide and very gently warmed. A slow current of oxygen is passed through a long tube 3 or 4 mm. in diameter whose end almost touches the surface of the liquid. A flame is then

brought to the mouth of the beaker, and soon the oxygen is seen burning at the end of the tube. The tube is then quickly immersed in the liquid and the current of oxygen simultaneously increased (Fig. 88). A green flame of considerable size appears in the beaker, and the oxygen is seen to be burning beneath the surface of the liquid. In a very few moments the ammonium hydroxide will have lost so much of the dissolved ammonia as to be too dilute for the continuation of the experiment.

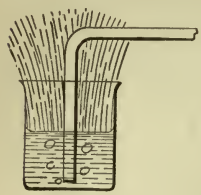

Fig. 88

Strongest NH_4OH ; O supply.

32. Decomposition by metallic potassium. — Potassium unites with ammonia, setting free some of the hydrogen. A 7 mm. piece of potassium is placed in a hard-glass bulb-tube and a gentle current of dry ammonia passed through it. On heating the bulb the potassium melts, and if heated strongly, the molten globule suddenly bursts, filling the bulb with the characteristic green vapor of potassium. The hydrogen set free may be ignited at the open end of the bulb-tube.

Bulb-tube ; K ; NH_3 supply.

33. Decomposition of ammonia by platinized asbestos. — When dry ammonia is passed over heated platinized asbestos in a combustion-tube, the gas is dissociated and three volumes of hydrogen and one of nitrogen are set free. The gases so liberated may be collected over water in the pneumatic trough and, owing to the large percentage of hydrogen, may be ignited.

Ammonia dried by passing through a U-tube filled with soda-lime or quicklime is conducted through a 25 cm. length of combustion tubing fitted with a cork and a delivery-tube

leading to the pneumatic trough. A 5 cm. length of platinized asbestos is placed in the middle of the combustion-tube and brought to a low red heat by means of a Bunsen burner. On conducting the dry ammonia through the tube all the gas will be absorbed in the pneumatic trough as soon as air has been driven out of the apparatus; but as the platinized asbestos begins to be heated, small quantities of gas, rapidly increasing with the increased temperature, will be collected at the pneumatic trough. On applying a match the gas will burn.

$$2\,NH_3 = N_2 + 3\,H_2.$$

25 cm. length combustion tubing; U-tube containing soda-lime or quicklime; platinized asbestos; NH_3 supply.

34. Electrolysis of ammonium hydroxide. — Ammonia is decomposed into the elements hydrogen and nitrogen when subjected to electrolysis.

Strong ammonium hydroxide, to which some sodium chloride has been added to give conductivity to the solution, is poured into the electrolytic apparatus (Fig. 46, p. 95) and decomposed by means of a current of from four to six cells. Platinum electrodes must be used. In a short time gas will be collected at both arms of the tube in the proportion of three volumes in one arm to one volume in the other. The larger volume of gas can be tested and shown to be hydrogen by opening the gas-cock and applying a match. The sodium chloride in the solution will, however, impart a yellowish color to the flame. The smaller volume of gas can be transferred to a test-tube in the manner shown in Fig. 89, and proved to be nitrogen by extinguishing a match.

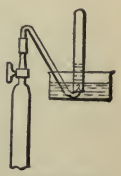

FIG. 89

$$2\,NH_3 = N_2 + 3\,H_2.$$

Electrolytic apparatus (Fig. 46, p. 95); battery 6 cells; con. NH_4OH; NaCl.

35. Quantitative decomposition of ammonia by chlorine. —Chlorine acts on ammonium hydroxide with the formation of hydrochloric acid and the liberation of nitrogen. Chlorine abstracts the hydrogen from ammonia, and consequently for every three volumes of chlorine used one volume of nitrogen is liberated.

The eudiometer (Fig. 11, p. 26) is filled over a salt solution with a rapid stream of pure chlorine. When the tube is completely filled, a well-fitting rubber stopper is crowded into the open end of the tube while still under the salt solution, care being taken to enclose no liquid in the tube. A few cubic centimeters of strongest ammonium hydroxide are then allowed to flow through the funnel into the gas, though the stop-cock must be but slightly turned. As the ammonia comes in contact with the chlorine, the reaction is very vigorous and, owing to the condensation in gas volume, a partial vacuum is produced inside the tube. The ammonium hydroxide in the bulb of the eudiometer is replaced by water acidulated with sulphuric acid, successive portions poured into the funnel and, by opening the stop-cock, allowed to flow into the eudiometer till the internal and the external pressure are the same, and no liquid will enter. The volume of colorless, residual gas will be found to be one-third the volume of the chlorine originally in the tube. The tube may be previously roughly graduated in thirds by rubber rings, allowance being made for the space occupied by the cork. A simpler method is to measure the distance from the base of the cork to the stop-cock in centimeters. The residual gas will measure one-third of this distance when the bulb is held in an upright position. By removing the cork the gas may be tested and shown to be nitrogen by extinguishing a splinter.

A glass tube, 1 m. long and 15 to 20 mm. in diameter, sealed at one end and fitted with a one-holed rubber stopper

through which the stem of a small dropping-funnel passes, may be used in case the eudiometer is not at hand. The tube is filled with chlorine, as above, inverted with the thumb over the mouth of the tube, and the cork on the stem of the dropping-funnel quickly thrust into the open end of the tube. The stem of the dropping-funnel should have been previously filled with strongest ammonium hydroxide. The funnel is then filled with the hydroxide, and a few cubic centimeters allowed to fall drop by drop into the gas. As each drop falls, it is seen to emit a feeble flash of light, a phenomenon not observable when the other form of eudiometer is used. The remaining operations are identical with those described above.

$$2\,NH_3 + 3\,Cl_2 = 6\,HCl + N_2.$$

$$NH_3 + HCl = NH_4Cl.$$

Eudiometer (Fig. 11, p. 26); Cl generator; glass tube sealed at one end, 1 m. long, 15–20 mm. diameter; dropping-funnel; rubber stoppers; strongest NH_4OH.

36. Decomposition of gaseous ammonia by sodium hypobromite and the volumetric relation of the nitrogen liberated. — The volumetric relation of the nitrogen liberated from a given volume of ammonia by the action of sodium hypobromite is easily shown by filling the eudiometer (Fig. 11, p. 26) with dry gaseous ammonia and introducing a strong solution of sodium hypobromite through the stop-cock. The eudiometer must be perfectly dry and is filled with ammonia, either by conducting the gas through a long glass tube thrust up into the eudiometer, which is clamped in a vertical position, or by conducting a stream of the dry gas through a cork inserted in the funnel bulb at the top. In the latter case the stop-cock is open and the air is driven out of the eudiometer at the lower opening. Where the

long glass tube is used to introduce the ammonia it must be withdrawn slowly so as to leave the eudiometer completely filled with the gas. A perfectly dry, well-fitting rubber stopper is then inserted in the bottom of the eudiometer and a strong solution of sodium hypobromite, made by adding 10 cc. of bromine to 50 cc. of strong sodium hydroxide solution, poured into the bulb. The stop-cock is carefully opened, and one drop of the hypobromite solution allowed to enter and come in contact with the gas. Inasmuch as there will probably be a slight pressure inside the tube, caused by the insertion of the rubber stopper, a bubble or two of the gas may escape. As soon as the liquid has entered the large volume of gas, rapid absorption takes place and the hypobromite solution will be drawn into the tube. Care must be taken to allow no air to enter, and accordingly the stop-cock must be closed before all the liquid has entered the eudiometer. A vigorous evolution of nitrogen will take place, and on filling the bulb with water and carefully opening the stop-cock, liquid will be drawn into the tube until it is half full. The residual gas may be tested by inverting the eudiometer, removing the stopper, and inserting a burning splinter. From two volumes of ammonia one volume of nitrogen is obtained.

$$2\,NH_3 + 3\,NaBrO = 3\,NaBr + 3\,H_2O + N_2.$$

Eudiometer (Fig. 11, p. 26); rubber stopper; supply of dry NH_3; NaBrO (10 cc. Br, 50 cc. NaOH sol.).

NITROGEN CHLORIDE

37. Preparation. — When chlorine reacts on an excess of ammonia, it combines with the hydrogen, liberating nitrogen (Ex. 35). If, however, an excess of chlorine is allowed to act on ammonia or ammonium chloride, a portion of

the chlorine unites directly with the nitrogen, forming a violently explosive, oily liquid, nitrogen chloride. The preparation of this body can be safely carried out only on a very small scale.

By electrolyzing a solution of ammonium chloride, chlorine is liberated at the positive pole, and in the nascent condition reacts with the excess of ammonium chloride, forming small quantities of nitrogen chloride. The electrolysis must be carried out in an open vessel (Fig. 36, p. 74) from which the graduated tubes have been removed. The electrodes, which are made by fusing pieces of platinum wire into bent glass tubes which are afterwards filled with mercury, are immersed in a strong solution of ammonium chloride, and the current from 4 or 5 cells of a bichromate battery should be passed through the solution. A millimeter layer of turpentine should be poured on the surface of the ammonium chloride solution, and it will be found that as the bubbles of chlorine ascend they will carry with them minute quantities of nitrogen chloride, which on coming in contact with the turpentine will explode. As the explosion may at times ignite the turpentine, a metal plate should be at hand with which to cover the dish and extinguish the flame. Occasionally small globules of nitrogen chloride will settle to the bottom of the dish; in that case it is best at the conclusion of the experiment to render the solution strongly alkaline with ammonium hydroxide and allow it to stand until the nitrogen chloride has of itself decomposed.

Electrolytic apparatus (Fig. 36, p. 74) ; battery.

NITROGEN IODIDE

38. Preparation. — (*a*) When iodine is allowed to act on strong ammonium hydroxide, a portion of the hydrogen of the ammonia is replaced by iodine, forming a mixture of

compounds containing nitrogen, hydrogen, and iodine, called nitrogen iodide. The direct precipitation of this compound is most easily effected by adding to a concentrated alcoholic solution of iodine an equal volume of the strongest ammonia water. The nitrogen iodide is immediately precipitated in the form of a black powder.

(*b*) The preparation of any quantity of nitrogen iodide is best secured by digesting powdered iodine with strongest ammonia for a few minutes. A few crystals of iodine are placed in a mortar and pulverized. Sufficient strong ammonia is added to cover the iodine completely, and the mixture is stirred from time to time. After a few minutes the mixture may be thrown on a filter-paper, washed with alcohol, and finally with water. The filter-paper is removed from the funnel and the still moist precipitate spread on several small pieces of filter-paper and laid on a porous plate to dry. Without the application of artificial heat the material will require about one hour to dry sufficiently to be explosive.

Porous plates; strongest NH_4OH; powdered I; alcohol.

39. Explosive character of nitrogen iodide. — The great sensitiveness of this compound to friction or heat is shown by the following experiments.

A piece of filter-paper on which a small quanity of nitrogen iodide has been dried is carefully placed on a table and then touched with a feather. Even this gentle friction is sufficient to cause it to explode.

A piece of paper containing nitrogen iodide is held in crucible tongs, and suddenly depressed over a Bunsen flame. The explosion is sufficent to blow out the gas.

A moistened filter-paper on which nitrogen iodide has been placed is stretched tightly over the mouth of a 7 cm. funnel. After drying, the nitrogen iodide may be exploded

with a feather. The explosion will blow a hole through the paper.

A dry paper containing some of the nitrogen iodide may be exploded on a moistened filter-paper stretched over the mouth of the funnel as above.

Nitrogen iodide may be exploded by a sudden puff of air. Air is blown from the lungs through a long glass tube upon dry nitrogen iodide on a piece of filter-paper. The nitrogen iodide is exploded. Successive papers may be blown on, and though the papers do not move, the iodide will be exploded.

A peculiar characteristic of the explosion produced by the nitrogen iodide is its inability to impart its explosion to a piece of dry guncotton. A small quantity of moist nitrogen iodide is laid on a small piece of guncotton. After the iodide has become thoroughly dry, it may be exploded by friction, but the guncotton will be unchanged. The combustible nature of the guncotton is shown by applying a flame.

Nitrogen iodide on filter-paper; guncotton; feather.

HYDROXYLAMINE

40. Reduction of copper sulphate solution. — The reducing action of hydroxylamine on an alkaline copper solution is shown by adding sodium hydroxide in excess to 1 drop of copper sulphate solution, diluting till nearly colorless, and adding a few drops of the hydroxylamine chloride solution. On heating the mixture, a yellow precipitate is obtained. This reaction serves as a very delicate test for hydroxylamine.

Hydroxylamine chloride solution; $CuSO_4$ solution.

41. Alternate reducing and oxidizing action on iron solutions. — A colorless solution of ferrous ammonium sulphate

is treated with sodium hydroxide, with the formation of the green ferrous hydroxide. On adding a few drops of hydroxylamine chloride solution to the ferrous hydroxide suspended in the alkaline liquid, the precipitate is immediately turned brown, indicating the formation of ferric hydroxide.

Hydroxylamine chloride, when acting on slightly acid solutions of ferric salts, effects their reduction, discharging the color of the solution. A portion of the suspended precipitate from the above experiment is treated with hydrochloric acid until completely dissolved, any great excess of acid being neutralized with sodium hydroxide. The solution must, however, have an acid reaction. On adding hydroxylamine chloride solution, the ferric salt is reduced and the solution becomes colorless.

Ferrous ammonium sulphate solution; hydroxylamine chloride.

NITROUS OXIDE

PREPARATION

42. By heating ammonium nitrate — Ammonium nitrate on heating is decomposed quantitatively into nitrous oxide and water.

Ten grams of the powdered salt are heated in a 100 cc. Jena glass Erlenmeyer flask fitted with a cork and a wide delivery-tube. The salt is somewhat hygroscopic, and hence it is advisable to dry it thoroughly by heating in an air-bath to 120°. A considerable quantity of the salt may be thus dried and preserved in well-stoppered bottles. On heating, the salt first melts and then decomposes, liberating nitrous oxide. As the liberation of the gas is likely to be somewhat violent, a wide delivery-tube is advisable. In heating, care should be taken to heat no more than is necessary to secure a regular flow of the gas. If the melted salt froths, or the

gas evolution is too great, the lamp should be removed, but replaced before water can rise in the delivery-tube and fall into the hot flask and break it. The explosive nature of the salt requires some care in its use, though if the previously dried salt is used, and the flame is turned very low, the experiment is very successful. The heating should be stopped before all the material is decomposed.

Owing to the solubility of nitrous oxide in cold water, the water in the pneumatic trough should be warmed to a temperature of between 30° and 40°. On the gradual application of heat, the gas is liberated, and may be collected as described. As no safety-tube is used in this experiment, it is advisable to remove the delivery-tube immediately after the gas has ceased to be evolved.

$$NH_4NO_3 = 2\ H_2O + N_2O.$$

100 cc. Jena glass Erlenmeyer flask; wide delivery-tube; pneumatic trough; warm water; dried NH_4NO_3.

PROPERTIES

43. Combustion of a splinter. — If a glowing splinter is thrust into a cylinder of nitrous oxide, it is immediately rekindled and burns with a degree of intensity similar to that of its combustion in pure oxygen.

Occasionally, in preparing the nitrous oxide, considerable quantities of free nitrogen are formed which diminish to a marked degree the activity of this gas in supporting combustion. The presence of nitrogen first effects the rekindling of a glowing splinter. If the gas relights the splinter, it is an indication of its purity. A burning splinter will, however, always burn with increased brilliancy in the gas, even if somewhat contaminated with nitrogen.

Cylinder of N_2O; splinter.

44. Combustion of hydrogen. — Hydrogen is allowed to burn from the recurved jet (Fig. 41, p. 85) and is then lowered into a cylinder of nitrous oxide. The flame undergoes a marked change in color, becoming covered with a blue envelope. The presence of nitrous acid after the combustion is shown by thrusting a piece of iodo-starch paper into the cylinder, where it is instantly turned blue.

Recurved jet (Fig. 41, p. 85); cylinder of N_2O; H supply; KI-starch paper.

45. Explosion of hydrogen and nitrous oxide. — A thick-walled cylinder is half filled over warm water with nitrous oxide, the remaining volume being filled with hydrogen. The cylinder is covered with a glass plate and a towel wrapped around it. On the application of a lighted taper an explosion occurs.

$$N_2O + H_2 = H_2O + N_2.$$

Cylinder; pneumatic trough filled with warm water; H generator; N_2O supply.

46. Combustion of sulphur. — Sulphur, burning feebly in a deflagrating-spoon, when lowered into a cylinder of nitrous oxide, is extinguished. If, however, it is strongly heated and brought almost to a boil before being introduced, it will burn brilliantly, forming sulphur dioxide.

$$S + 2\,N_2O = SO_2 + 2\,N_2.$$

Jar of N_2O; sulphur in deflagrating-spoon.

47. Combustion of phosphorus. — A small piece of phosphorus is placed in the deflagrating-spoon, and after being ignited is quickly thrust into a jar of nitrous oxide. The combustion, similar to that in pure oxygen, continues with great brilliancy.

Jar of N_2O; P.

48. Combustion of iron. — A cylinder or tall beaker, having a piece of asbestos paper or wire gauze in the bottom, is filled with nitrous oxide. A bundle of iron wires, tipped with a bit of molten sulphur, is ignited and lowered into the jar. The combustion proceeds with great brilliancy, the particles of molten iron oxide falling to the bottom and striking on the asbestos. It is advisable to have a centimeter layer of water on the bottom of the cylinder.

Cylinder with asbestos in bottom, filled with N_2O; bundle of iron wires tipped with sulphur.

NITRIC OXIDE

PREPARATION

49. From nitric acid and copper. — The action of copper on dilute nitric acid results in the formation of nitric oxide.

To prepare this gas, copper clippings are placed in a 500 cc. Erlenmeyer flask fitted with a thistle-tube and a delivery tube, the thistle-tube extending to the bottom of the flask. The copper is covered with water, and concentrated nitric acid added through the thistle-tube. The gas is evolved without the aid of heat and may be collected at the pneumatic trough. If the gas evolution is too violent, water is added through the thistle-tube to dilute the acid. If, on the other hand, the flow of gas is slow, concentrated nitric acid is added. After the reaction has been running for a few minutes the gaseous contents of the flask become colorless, while any of the gas escaping into the air has a deep reddish brown color. The cylinders of gas collected at the pneumatic trough are also colorless.

Provision must be made for conducting the excess of gas into a hood, as the fumes are very irritating.

$$3\,Cu + 8\,HNO_3 = 3\,Cu(NO_3)_2 + 4\,H_2O + 2\,NO.$$

500 cc. Erlenmeyer flask; Cu clippings; thistle-tube and glass elbow.

50. By the action of sulphuric acid on copper and potassium nitrate solution. — Instead of adding nitric acid directly to copper, the acid may be formed by adding sulphuric acid to a strong solution of a nitrate covering a quantity of copper clippings.

Copper clippings in the bottom of an Erlenmeyer flask are covered with a saturated solution of potassium nitrate. Concentrated sulphuric acid is allowed to drop from a dropping-funnel upon the liquid. A steady evolution of nitric oxide is obtained, the rate of which is determined by the dropping of the sulphuric acid.

$$2\,KNO_3 + H_2SO_4 = K_2SO_4 + 2\,HNO_3.$$

$$3\,Cu + 8\,HNO_3 = 3\,Cu(NO_3)_2 + 4\,H_2O + 2\,NO.$$

Apparatus (Fig. 3, p. 11); Erlenmeyer flask; dropping-funnel; saturated solution of KNO_3; Cu clippings.

51. From sodium nitrite and acidulated ferrous chloride. — When a strong solution of sodium nitrite is allowed to drop into a solution of ferrous chloride to which hydrochloric acid has been added, a regular evolution of nitric oxide is obtained.

A handful of iron nails is placed in a 500 cc. Erlenmeyer flask and covered with 200 cc. of concentrated hydrochloric acid, and the flask is allowed to stand over night. Sufficient ferrous chloride will have been formed during this time to give a strong evolution of nitric oxide with sodium nitrite. A two-holed rubber stopper fitted with a dropping-funnel containing a saturated solution of sodium nitrite and a delivery-tube is inserted in the neck of the flask.

500 cc. Erlenmeyer flask; dropping-funnel; cork and delivery-tube; iron nails; saturated solution of $NaNO_2$.

PROPERTIES

52. Neutrality of pure nitric oxide to litmus. — A stream of nitric oxide is conducted through a clean delivery-tube under a cylinder inverted over a crystallizing dish of water. The water should be neutral and the cylinder should not be completely filled, the supply of nitric oxide being cut off that it may not come in contact with the air, thereby turning the liquid slightly acid. A piece of blue litmus paper fastened to the end of a copper wire is then pushed up into the interior of the flask, care being taken that no air is admitted. No change will be observed in the blue litmus until a small quantity of air is admitted. The nitrogen peroxide formed will then turn the litmus red.

NO generator; cylinder and crystallizing dish; pure neutral water; blue litmus paper; copper wire.

53. Solubility in ferrous sulphate solution. — Nitric oxide is collected in the eudiometer (Fig. 11, p. 26), and ferrous sulphate solution allowed to flow slowly through the gas by opening the stop-cock. The nitric oxide is rapidly absorbed, the solution becoming a dark brown.

To prepare a considerable quantity of a solution of nitric oxide in ferrous sulphate, the gas is conducted into a gas washing-bottle containing a solution of ferrous sulphate or ferrous ammonium sulphate (Mohr's salt). In case the latter is used, the color change is more noticeable; for the salt solution before the introduction of the gas is colorless, while as the gas is absorbed, the color passes rapidly to a very dark brown.

On heating the liquid, nitric oxide is again liberated.

Eudiometer (Fig. 11, p. 26); gas washing-bottle; NO generator; $FeSO_4$ solution; $(NH_4)_2Fe(SO_4)_2$ solution.

54. Absorption by nitric acid. — (*a*) Nitric oxide is absorbed by nitric acid, with the formation of nitrous acid and nitrogen peroxide. The variation in the nature of the products formed corresponds to the variations in strength of the nitric acid used.

Three gas washing-bottles are connected in series and nitric oxide allowed to pass through them. The first bottle contains nitric acid of a specific gravity of 1.25; the second, nitric acid of the specific gravity of 1.35; and the last, acid of the specific gravity of 1.5. The gas issuing from the last bottle is then conducted into a flue. On allowing the nitric oxide to pass through this system for some time, a series of color changes is observed in the different bottles. The first bottle contains nitrous anhydride and nitrous acid, and will acquire a blue color, especially if cooled in a beaker of ice-water. The second will possess a green color and the last the deep brown color of fuming nitric acid.

Three gas washing-bottles; NO generator; HNO_3 of 1.25, 1.35, and 1.5 specific gravities; ice.

(*b*) Nitric oxide is collected in the eudiometer (Fig. 11, p. 26) over water and concentrated (not fuming) nitric acid is allowed to flow down into the gas through the stop-cock. The absorption is quite rapid, as is indicated by the rise of the liquid in the tube.

Eudiometer (Fig. 11, p. 26); NO generator; con. HNO_3.

55. Absorption by acidulated potassium permanganate solution. — Nitric oxide is oxidized to nitric acid by potassium permanganate in the presence of sulphuric acid. The absorption of the gas and the decolorization of the permanganate solution may be shown by collecting a quantity of the nitric oxide in the eudiometer (Fig. 11, p. 26) and allowing a solution of potassium permanganate, strongly

acidulated with sulphuric acid, to flow slowly down through the gas.

$$6\,KMnO_4 + 9\,H_2SO_4 + 10\,NO = 10\,HNO_3 + 3\,K_2SO_4 + 6\,MnSO_4 + 4\,H_2O.$$

Eudiometer (Fig. 11, p. 26) ; NO supply ; $KMnO_4 + H_2SO_4$.

56. Nitric oxide and air. — A tall glass cylinder filled with nitric oxide and covered with a glass plate is placed mouth upwards on the table. A cylinder filled with air is placed mouth downward on top of the glass plate covering the cylinder of nitric oxide. On slipping out the glass plate between the two cylinders red fumes appear where the air and nitric oxide come in contact. As the nitrogen peroxide formed is much heavier than either air or nitric oxide, the experiment is especially interesting in showing the diffusibility of the gases.

Cylinder of NO ; empty cylinder.

57. Union with pure oxygen. — Nitric oxide unites with pure oxygen quantitatively, forming nitrogen peroxide. In this union a contraction takes place, as two volumes of nitric oxide combine with one volume of oxygen to form two volumes of nitrogen peroxide.

A liter bottle is fitted with a two-holed rubber stopper carrying a straight glass tube extending to within 7 cm. of the bottom of the bottle and drawn out to a jet 2 mm. in diameter. A glass elbow is thrust through the second hole of the rubber stopper. Rubber tubes and pinch-cocks are slipped on the ends of both glass tubes, and the rubber stopper and fittings thrust under water into the mouth of the bottle, which has previously been three-fourths filled with nitric oxide at the pneumatic trough (Fig. 90). Both pinch-cocks being closed, the apparatus is supported mouth

downwards, and a glass tube dipping into a beaker of blue litmus solution is connected with the rubber tube connected with the jet in the bottle. Oxygen under pressure, preferably from a cylinder of the compressed gas, is forced through the glass elbow after opening the pinch-cock until a few bubbles have entered the bottle. As each bubble of the oxygen comes in contact with the nitric oxide, red fumes of nitrogen peroxide will appear. These are soon dissolved by the water remaining in the bottle, and the gas again becomes clear. The process is repeated a number of times, and finally the pinch-cock connecting the glass jet with the blue litmus solution is opened. On account of the vacuum inside the flask caused by the solubility of nitrogen peroxide in water, the blue litmus solution will rise rapidly through the jet and enter the flask in the form of a fountain. There in the presence of acid fumes the color will be turned from blue to red.

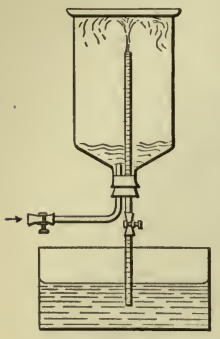

FIG. 90

Apparatus (Fig. 90) ; liter bottle ; rubber stopper (2-holed) ; pinch-cocks and tubes ; O supply ; blue litmus solution.

58. Combustion in nitric oxide. — A glowing or burning splinter when thrust into nitric oxide is extinguished.

A burning candle on being lowered into nitric oxide is extinguished.

Burning sulphur on a deflagrating-spoon is extinguished when lowered into the gas.

Phosphorus when feebly burning on a deflagrating-spoon is likewise extinguished when lowered into nitric oxide.

If, however, a piece of phosphorus is placed on a deflagrating-spoon and allowed to burn vigorously in the air and

is then lowered into the nitric oxide, the heat generated by its combustion is sufficient to dissociate the gas, and consequently the combustion proceeds even more vigorously than in air.

Jars of NO ; deflagrating-spoons ; candle ; P ; S.

59. Combustion of charcoal.— A piece of charcoal fastened to a stout iron wire is heated to glowing in a Bunsen flame. On thrusting it into a cylinder of nitric oxide the flame is extinguished.

If, however, the charcoal is placed in a bulb-tube and strongly heated with a Bunsen flame, it will glow in a current of nitric oxide. Provision should be made for conducting the escaping gas into a flue.

NO generator ; jar of NO ; bulb-tube ; charcoal.

60. Combustion of carbon disulphide. — When the vapor of carbon disulphide is mixed with nitric oxide and the mixture ignited, a characteristic blue flame is produced which has great actinic power, and has consequently been used as artificial illumination for taking photographs.

Three or four cubic centimeters of carbon disulphide are rapidly poured from a test-tube into a 250 cc. cylinder of nitric oxide covered with a plate. The glass plate is slipped to one side to permit the introduction of the carbon disulphide, and then the cylinder is immediately closed. The cylinder is then thoroughly shaken to mix the vapors, the glass plate removed, and the mixture ignited.

A blue flame results, the sulphur being deposited on the sides of the vessel. The cylinder should be immediately washed, as otherwise the sulphur will be hard to remove.

Jar of NO ; CS_2.

NITROUS ANHYDRIDE AND NITROUS ACID

61. Preparation of nitrous anhydride from arsenic trioxide and nitric acid. — When arsenic trioxide is oxidized by nitric acid of a specific gravity of from 1.3 to 1.35, gaseous nitrous anhydride is liberated.

Twenty-five grams of arsenic trioxide are placed in a 250 cc. flask fitted with a thistle-tube and an elbow. The arsenic is covered with nitric acid of the above-mentioned specific gravity and the mixture gently warmed. The gas is heavy, and may be collected by displacement in a large flask. When collected in this manner, the deep reddish brown color is very noticeable.

$$2\ As_2O_3 + 4\ HNO_3 + 4\ H_2O = 4\ H_3AsO_4 + 2\ N_2O_3.$$

250 cc. flask; thistle-tube and elbow; large flask; HNO_3 specific gravity 1.3–1.35; As_2O_3.

62. Liquefaction of nitrous anhydride. — The gaseous nitrous anhydride obtained from the preceding experiment is passed successively through a gas washing-bottle immersed in a beaker of cold water to condense the steam formed by the reaction, and then through a U-tube packed in a freezing-mixture of ice and salt. The water in the first beaker must not be below 8° C. On removing the U-tube from the freezing-mixture a blue liquid will be found; but as some nitrogen peroxide often condenses with it, the color is not always clear. On the addition of a small quantity of ice-water, the color immediately appears perfectly blue. The liquid boils readily on the application of a very gentle heat.

N_2O_3 supply; gas washing-bottle; U-tube; freezing-mixture of ice and salt.

63. Phenylenediamine reaction for nitrous acid. — Phenylenediamine solution, when added to a solution of sodium nitrite, to which a few drops of sulphuric acid have been added, gives an intense orange-yellow color.

The delicacy of this reaction is so great as to make its use of great importance in determining minute quantities of nitrites in potable waters. A few drops of acidulated sodium nitrite solution are added to 2 l. of water in a large beaker. On the addition of a few drops of phenylenediamine solution, a distinct yellow coloration will appear even at this great dilution.

$NaNO_2$; phenylenediamine solution.

NITROGEN PEROXIDE

PREPARATION

64. By ignition of lead nitrate. — Lead nitrate on ignition is decomposed into lead monoxide, nitrogen peroxide, and oxygen. Nitrogen peroxide, being easily condensed, is retained in a U-tube packed in salt and ice, while the oxygen is collected at the pneumatic trough.

Fifty grams of previously dried lead nitrate are placed in a 200 cc. Jena glass distilling flask, connected to one limb of a U-tube packed in ice and salt; the other limb is fitted with a cork and a delivery-tube leading to the pneumatic trough (Fig. 91). The neck of the distilling flask is securely corked, and the lead nitrate carefully heated with a Bunsen burner. After all the air has been driven out of the apparatus, the oxygen may be collected and tested at the pneumatic trough. On disconnecting the apparatus, the impure nitrogen peroxide will be found as a red liquid condensed in the U-tube. The

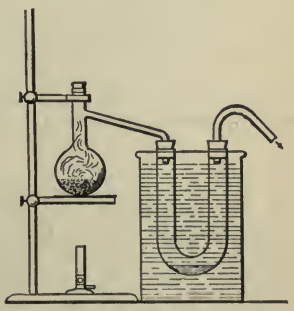

Fig. 91

lead nitrate used for this experiment should be dried by heating in a porcelain evaporating-dish till red fumes just appear.

200 cc. Jena glass distilling-flask ; U-tube in freezing-mixture ; dried pulverized $Pb(NO_3)_2$.

65. By the action of tin on concentrated nitric acid. — Tin and nitric acid react vigorously, nitrogen peroxide being evolved.

The bottom of a 300 cc. Erlenmeyer flask is covered with granulated tin, and strong nitric acid is introduced through a thistle-tube reaching to the bottom of the flask. A glass elbow thrust through the cork in the neck of the flask is connected to a gas washing-bottle, which is in turn connected with a U-tube immersed in a freezing-mixture of salt and ice. As soon as the acid is added, the reaction begins and great heat is developed. Most of the steam formed is condensed in the gas washing-bottle. The nitrogen peroxide is condensed in the U-tube. By disconnecting the U-tube and connecting a glass tube to the gas washing-bottle, the gas may be collected by displacement.

$$5\ Sn + 20\ HNO_3 = Sn_5O_5\,(OH)_{10} + 5\ H_2O + 20\ NO_2. \quad [?]$$

300 cc. Erlenmeyer flask ; thistle-tube and glass elbow ; 2-holed cork ; gas washing-bottle ; U-tube ; freezing-mixture ; Sn.

PROPERTIES

66. Vaporization of liquid nitrogen peroxide. — A few drops of liquid nitrogen peroxide are placed in a flask, the bottom of which is warmed with the hand. The liquid immediately evaporates, filling the flask with deep brownish red fumes.

250 cc. flask ; liquid NO_2.

67. Dilution of fuming nitric acid. — On diluting fuming nitric acid with water the excess of nitrogen peroxide in the nitric acid undergoes decomposition in the presence of water, forming nitrous anhydride, the various steps in the dilution being characterized by color changes in the solution.

Twenty cubic centimeters of fuming nitric acid in a beaker are gradually diluted with water. The reddish brown acid becomes light yellow and then green. If the green solution is cooled with ice, a blue color will be obtained.

The reverse of this operation may be carried out by gradually adding to some crushed ice in a beaker fuming nitric acid. In this case the solution becomes first blue, then green, light yellow, and finally dark brown.

Fuming HNO_3; ice.

68. Combustion of charcoal. — Glowing charcoal when in contact with liquid nitrogen peroxide burns brilliantly in the oxygen liberated from this compound.

Two cubic centimeters of liquid nitrogen peroxide are placed in a test-tube clamped in a vertical position. A piece of charcoal fastened to one end of an iron wire and brought to a glow in a Bunsen flame is carefully lowered till it just touches the surface of the liquid. It continues to burn with increased brilliancy.

Liquid NO_2; piece of charcoal.

69. Combustion of potassium in gaseous nitrogen peroxide. — Potassium combines with the oxygen of nitrogen peroxide, burning with considerable brilliancy.

A 3 mm. piece of potassium is placed in a deflagrating-spoon, slightly warmed, and lowered into a cylinder of nitrogen peroxide having a layer of sand at the bottom. The potassium takes fire and burns brilliantly.

Jar of NO_2; deflagrating spoon; K.

70. Absorption of nitrogen peroxide by sulphuric acid. (Formation of chamber crystals.) — The formation of nitrogen peroxide and the subsequent absorption of this compound by sulphuric acid is shown by conducting nitric oxide from a suitable generator into a large flask, the sides of which have been moistened with concentrated sulphuric acid. A two-holed rubber stopper in the neck of the flask contains a glass tube extending halfway to the bottom of the flask and a short glass elbow which is connected with the draft or flue.

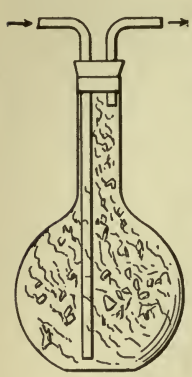

FIG. 92

Nitric oxide is conducted through the long glass tube into the flask, where it combines with the oxygen of the air, forming the deep red fumes of nitrogen peroxide, which are immediately absorbed by the sulphuric acid. The whole interior of the flask becomes covered with a crystalline deposit of chamber crystals (Fig. 92).

Large flask (2-6 l.); NO generator.

NITRIC ACID

PREPARATION

71. From potassium nitrate and sulphuric acid. — The general principle of using a strong acid to drive a weaker out of combination is made use of in the preparation of nitric acid. Thirty grams of powdered potassium nitrate are placed in a 500 cc. glass-stoppered retort and 18 cc. of concentrated sulphuric acid are carefully poured through a funnel upon the mixture, care being taken to prevent any acid from getting into the neck of the retort. The retort is then gently agi-

tated to insure a thorough mixture of the ingredients. It is then clamped so that it may be heated on a wire gauze, its neck being thrust deep into a 500 cc. flask partially immersed in water in a large crystallizing-dish (Fig. 93). On applying a very gentle heat, nearly colorless nitric acid distils over and condenses in the flask.

$$KNO_3 + H_2SO_4 = KHSO_4 + HNO_3.$$

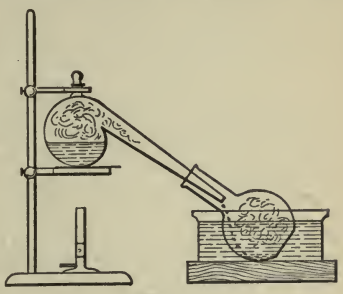

FIG. 93

500 cc. glass-stoppered retort; 500 cc. flask; crystallizing-dish; KNO_3.

PROPERTIES

72. Intense acid reaction. — Nitric acid possesses a strong acid reaction which is readily shown by dipping a glass rod into fuming nitric acid and then into a beaker containing a liter of water. Even at this great dilution the acidified water will instantly redden a strip of blue litmus paper.

73. Action on organic matter. — Nitric acid attacks organic matter, especially that of an animal nature, staining it yellow. The simplest example of this property of nitric acid is the familiar fact that the acid stains the fingers yellow. A large white feather has approximately the same composition as the human skin, and when dipped into fuming nitric acid in a beaker is stained a brilliant yellow, while a considerable portion of the feather is actually destroyed. On washing off the excess of acid and dipping the feather into dilute ammonium hydroxide, the color is somewhat intensified and becomes fixed.

Acid spots on clothing, if caused by sulphuric or hydro-

chloric acid, are easily effaced by moistening with ammonium hydroxide. Spots resulting from the action of nitric acid, however, cannot be removed.

Large white feather ; fuming HNO_3.

74. Action on turpentine. — Strongest nitric acid oxidizes turpentine, even in the cold, with such rapidity as to ignite the hydrocarbon.

A small evaporating dish is placed on a layer of sand in the bottom of a 1 or 2 l. beaker and one-third filled with a mixture of equal volumes of fuming nitric acid and concentrated sulphuric acid. This mixture is easily secured by slowly pouring 5 cc. of concentrated sulphuric acid into an equal volume of fuming nitric acid held in a test-tube, keeping the test-tube cool if necessary under the water tap, though care should be taken not to get any water in the mixture of acids. A few drops of turpentine are drawn up into a long (1 m.) glass tube with an elbow at one end, the short arm (15 cm.) of which is drawn out to a point (Fig. 50, p. 102). By raising the thumb from the open end of the tube the turpentine is allowed to drop on the acid mixture in the evaporating-dish. As each drop comes in contact with the acid, it bursts into flame. Care should be taken to prevent any drops of acid from flying out of the beaker, and provision should be made for carrying off the vapors rising from the combustion.

2 l. beaker ; small evaporating-dish ; long bent glass tube ; fuming HNO_3 ; conc. H_2SO_4 ; turpentine.

75. Action on sawdust. — Sawdust is heated in a small evaporating-dish till it is just about to char, and 3 cc. of fuming nitric acid are poured upon the mass. The carbonaceous material is immediately oxidized and the mixture undergoes a vigorous combustion.

76. Action on sugar. — Nitric acid does not act upon sugar in the cold, as may be seen by pouring 5 cc. of the acid over 2 g of sugar in the bottom of a test-tube. On warming the mixture, however, a violent reaction takes place, the sugar being oxidized

77. Action on wood, carbon disulphide, and illuminating gas. — The vigorous oxidizing action of nitric acid is best shown when the acid is freshly prepared by heating a mixture of potassium nitrate and sulphuric acid. Such a mixture is heated in a small wide-mouthed flask and a glowing splinter thrust into the liquid. The wood is instantly ignited and burns brightly.

If a few drops of carbon disulphide are poured from a test-tube into the flask and a flame held instantly at the mouth, combustion ensues in a most brilliant manner, the carbon disulphide burning with a deep blue flame.

A slow stream of illuminating gas passing through a long bent glass tube is ignited and thrust into the neck of the flask. Combustion proceeds vigorously.

78. Combustion of illuminating gas in fuming nitric acid. — Fuming nitric acid, containing, as it does, an excess of the oxides of nitrogen, supports the combustion of illuminating gas even when the end of the tube conducting the burning gas is thrust beneath the surface of the acid.

Fuming nitric acid is placed in a beaker in a strong draft. A flame of illuminating gas about 2 cm. long is allowed to burn at the end of a long glass elbow, and on thrusting the burning flame under the surface of the acid the combustion will continue.

Beaker; long glass elbow; fuming HNO_3.

79. Decomposition of nitric acid by heat. — At a high temperature nitric acid undergoes decomposition, with the formation of nitrogen peroxide and oxygen. A very simple and efficient method for showing this decomposition consists in allowing the nitric acid in vapor form to pass over pumice-stone heated in a piece of combustion tubing 40 cm. long and 15 mm. in diameter, preferably of Jena glass (Fig. 94). A thick piece of rubber tubing is slipped over one arm of a large glass elbow 10 mm. in diameter which is then crowded into one end of the combustion-tube, thereby making a tight joint. In the other end of the glass elbow a dropping-funnel con-

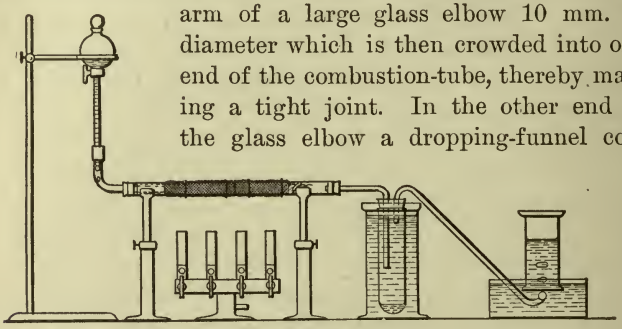

FIG. 94

taining strong nitric acid is fitted by thrusting the stem of the funnel through a piece of rubber tube slipped over the end of the elbow.

A roll of previously ignited asbestos paper 6 cm. long is inserted in the combustion-tube and so arranged that the glass elbow is pushed a short distance into its core. The rest of the tube is filled with broken bits of pumice-stone and the end closed with a cork carrying a small glass elbow, the other end of which is thrust some distance through a two-holed rubber stopper. The rubber stopper is inserted in the mouth of a large test-tube, kept cool by being immersed in water, and a delivery-tube connects the test-tube with the pneumatic trough. The combustion-tube is first strongly heated over a four-tube burner and concentrated

nitric acid then allowed to drop slowly (10 to 15 drops per minute) into the glass elbow. If the tip of the dropping-funnel is below the rubber connection, the rate of dropping may be easily determined. The nitric acid flows down the glass elbow and comes in contact with the asbestos coil. This, acting as a wick, conducts the liquid toward the warmer portion of the tube, where it is gradually vaporized. The vapor then passes over the heated pumice-stone and there undergoes decomposition. The water and nitrogen peroxide condense in the test-tube and the oxygen may be collected at the pneumatic trough. If the burners are so arranged that the asbestos roll is heated only at one end, *i.e.*, that nearest the pumice-stone, by placing a burner directly under this end of the asbestos, a regular gradation of temperature is secured along the asbestos roll, which is very hot at the inner end and cool at the end nearer the cork. A roll of wire gauze may be wound around the outside of that portion of the combustion-tube which is occupied by the pumice-stone and consequently requires the highest heat. By use of the asbestos roll the contact of the liquid with hot glass is avoided, and the glass tube is therefore seldom broken. After considerable use the rubber connections in each end of the combustion-tube are destroyed by the action of the strong nitric acid.

By allowing the acid to drop continually at a very slow rate the dropping-funnel acts as a safety-tube; for should the pressure inside the tube be materially increased, the dropping of the acid would cease. The dropping-funnel should, however, have a constricted tip of not more than 3 mm. in diameter, and not more than 10 cc. of acid should be placed in the dropping-funnel at a time.

$$4\ HNO_3 = 2\ H_2O + 4\ NO_2 + O_2.$$

Combustion-tubing, Jena glass; dropping-funnel; large glass elbow; 4-tube burner; pumice-stone; asbestos paper; large test-tube; pneumatic trough and cylinders.

80. Test for nitric acid. — The brown ring produced by the action of a solution of ferrous sulphate on concentrated sulphuric acid containing nitric acid is strikingly shown by placing 150 cc. of sulphuric acid containing a small quantity of potassium nitrate in a 500 cc. cylinder and allowing a concentrated solution of ferrous sulphate to flow from a dropping-funnel through a glass tube which delivers the liquid on the surface of the sulphuric acid solution (Fig. 95). In this way the two liquids are introduced into the cylinder without unnecessary mixing, and a deep brown ring is formed at their surface of contact.

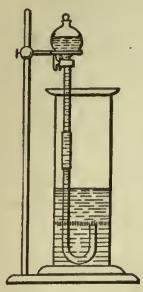

FIG. 95

500 cc. cylinder; con. $H_2SO_4 + KNO_3$; $FeSO_4$ solution.

HYDRAZINE

81. Reducing action on solutions of copper and silver. — Hydrazine sulphate is a strong reducing agent, causing the reduction of alkaline solutions of copper and silver salts.

A solution of cupric sulphate to which tartaric acid and potassium hydroxide have been added is treated with a solution of hydrazine sulphate. Even in the cold some reduction is obtained, and on warming a little, cuprous oxide is precipitated.

A solution of silver nitrate to which has been added sufficient ammonium hydroxide to redissolve the precipitate first formed is treated with a solution of hydrazine sulphate, which, on warming, effects the reduction with a deposition of finely divided silver.

Solutions of hydrazine sulphate, $AgNO_3$, $CuSO_4$ and tartaric acid.

82. Action with potassium iodate. — Hydrazine sulphate reduces potassium iodate, precipitating iodine, and in the process of the reaction the hydrazine is decomposed, liberating nitrogen.

A strong solution of potassium iodate is treated with a solution of hydrazine sulphate. A vigorous evolution of nitrogen is obtained, and the mixture becomes colored with liberated iodine. If the two solutions are hot, the iodine is vaporized and escapes from the mouth of the tube as a violet vapor. A drop of starch solution produces the characteristic blue color with the iodine.

$$15\,N_2H_4\,.\,H_2SO_4 + 12\,KIO_3 = 15\,N_2 + 36\,H_2O + 6\,K_2SO_4 + 9\,H_2SO_4 + 6\,I_2.$$

Hydrazine sulphate ; KIO_3 ; starch solution.

83. Decomposition of hydrazine sulphate by heat. — Hydrazine sulphate decomposes on heating, with the liberation of sulphur dioxide, hydrogen sulphide, and free sulphur. A few crystals of the salt are heated in a test-tube and the issuing gas tested for sulphur dioxide and hydrogen sulphide. Free sulphur will be deposited on the sides of the tube.

On dissolving the residue in water and adding a few drops of sulphuric acid sulphur will be precipitated.

HYDRAZOIC ACID

84. Preparation. — Nitric acid, of a specific gravity of 1.3, when allowed to act on hydrazine sulphate, gives a very good yield of hydrazoic acid.

One and one-half grams of hydrazine sulphate are placed in a test-tube fitted with a cork carrying a glass tube doubly bent so as to dip into a second test-tube containing silver

nitrate solution (Fig. 96). Four cubic centimeters of nitric acid of a specific gravity of 1.3 are added to the hydrazine sulphate, the cork inserted, and the mixture very slightly warmed. As the temperature rises, the gas will be evolved and bubble through the silver nitrate solution, producing a curdy white precipitate of silver nitride. A piece of moistened litmus paper, held in the mouth of the test-tube at the end of the operation, shows the acid character of the gas. The silver nitride formed should be filtered off, thoroughly washed with water, and allowed to stand till ready for use in the following experiments. Owing to its dangerously explosive nature but small quantities must be dried at a time.

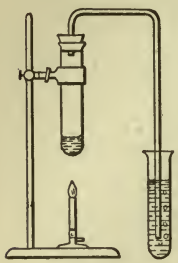

FIG. 96

Hydrazine sulphate; HNO_3 (1.3 specific gravity); $AgNO_3$ solution.

85. Preparation of hydrazoic acid (silver nitride) from hydrazine sulphate and silver nitrite. — Silver nitrite reacts with a solution of hydrazine sulphate, forming silver nitride.

Twenty-five cubic centimeters of a saturated solution of hydrazine sulphate in water are placed in a 50 cc. flask to which silver nitrite in small quantities is gradually added. The reaction is quite marked, the white, curdy silver nitride remaining suspended in the liquid. The precipitate should be filtered and dried as above. But very small quantities should be prepared at one time.

$$N_2H_4 + AgNO_2 = AgN_3 + 2H_2O.$$

Hydrazine sulphate; $AgNO_2$.

86. Explosive nature of silver nitride. — The great explosibility of silver nitride necessitates that experiments on this compound be carried out on a very small scale only. A

minute quantity (2 or 3 mg.) is heated on a knife-blade thrust into the Bunsen burner. The explosion is very great.

If a minute quantity of the precipitate is placed in the centre of a thin copper sheet which is then suddenly thrust into the flame, the force of the explosion is such as to make a dent in the copper.

Silver nitride is equally sensitive to friction, and a small piece placed between paper on an anvil and struck with a hammer detonates with great violence.

Hammer and anvil; dry AgN_3 (Care !!!); thin sheet copper.

PHOSPHORUS

PHOSPHORUS

MANIPULATION

1. Precautions in handling phosphorus. — Owing to its inflammable nature and the dangerous character of the wounds resulting from its burns, phosphorus must be handled with the greatest care. It may be handled beneath water with perfect impunity, though too much care cannot be exercised in avoiding danger from particles adhering to the fingers and finger nails. In all operations where phosphorus is to be removed from the water it should never be touched with the naked fingers, but with pincers or crucible tongs. Small pieces can be cut from a piece of phosphorus by submerging the knife and hand as well as the phosphorus in water. A more satisfactory method of obtaining small pieces is to use the globules as prepared in Ex. 4, or to break off pieces of the desired length from sticks of small calibre prepared as in Ex. 3. In drying a piece of phosphorus between filter-paper the greater portion of the water should be allowed to drain off by capillary attraction, and then the paper should be folded once over the phosphorus and gently pressed with the fingers. Under no circumstances should the fingers come in direct contact with the phosphorus. In cutting phosphorus it is important that the vessel used

should be thick, so as not to be broken by the accidental slipping of the knife.

Phosphorus is always kept under water, and care should be taken that the phosphorus after long standing does not become uncovered by the evaporation of the water in the vessel.

Wounds resulting from burns of phosphorus demand special and immediate treatment. They are very persistent and slow to heal, often resulting in running sores of a serious nature. The burn should immediately be washed in water and moistened with sodium bicarbonate or a dilute solution of bleaching-powder.

Burning phosphorus should be extinguished as directed in Ex. 8.

2. Melting and casting phosphorus. — Commercial phosphorus is ordinarily obtained in the form of sticks. Since after keeping for some time, especially on exposure to the light, the phosphorus acquires a dark color, it is desirable to show the translucent, waxy appearance of pure phosphorus by melting the commercial material and recasting it in sticks of convenient size.

Several sticks of phosphorus are placed in a beaker and completely covered with water. The beaker is then placed in an evaporating-dish partially filled with water, which is brought nearly to the boiling point. As the water in the beaker becomes warm, the phosphorus melts and settles to the bottom of the beaker. The phosphorus is best cast in small thin-glass test-tubes, which are filled with water and immersed in a

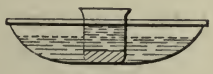

FIG. 97

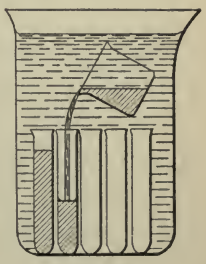

FIG. 98

large beaker of warm water. If the beaker in which the phosphorus has been melted is small enough, it also may be immersed in the water in the large beaker and the phosphorus poured under water into the test-tubes (Fig. 98). It is necessary to leave a centimeter layer of water on top of the phosphorus in each test-tube.

The molten phosphorus may be drawn up into a pipette and then allowed to run into the test-tubes. A 25 cc. pipette with a rather wide delivery-tip is immersed in a tall cylinder filled with warm water (Fig. 99). The pipette is then withdrawn, allowing the water to flow out, and while still warm dipped into the beaker containing the melted phosphorus. A layer of water is first drawn into the pipette and then the end dipped beneath the molten phosphorus. The pipette is nearly filled with phosphorus and then transferred to the beaker holding the test-tubes. As in this transference a few drops of melted phosphorus are liable to fall out of the tip of the pipette, it is best to seal the tip by dipping it into a deflagrating-spoon filled with water. The pipette should occasionally be dipped in the water in the tall cylinder to keep it warm enough to prevent the phosphorus from solidifying. After a few test-tubes are filled they may be removed from the beaker and placed in a test-tube rack, the layer of water on top preventing oxidation. After a few minutes the contents of the tubes will have solidified, and by making a hole in the bottom of each tube the stick of phosphorus may be forced out under water. Made in this manner, the sticks show the true color of the yellow phosphorus, and should be preserved under water in the dark. If care is

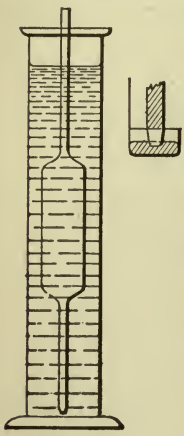

FIG. 99

always taken to draw a little water into the pipette before the phosphorus and to keep the tip of the pipette always under the phosphorus or water, the suction may be applied with the mouth, though it should always be borne in mind that carelessness will result in drawing melted phosphorus into the mouth. A safer method of operating is to insert a gas washing-bottle containing a small amount of water between the mouth and the pipette. The air should be drawn through the glass tube whose end terminates just below the cork. If any phosphorus should accidentally be drawn over, it would pass through the long tube extending to the bottom of the bottle, and there be delivered under water.

Large evaporating-dish ; tall cylinder ; gas washing-bottle ; 25 cc. pipette ; deflagrating-spoon ; thin test-tubes ; P.

3. Preparation of stick phosphorus. — Phosphorus may also be obtained in the stick form by drawing the molten liquid up into a glass tube of the diameter of the sticks desired.

A glass tube, from 8 to 10 mm. internal diameter and 20 cm. long, is provided with a one-holed cork or rubber connection which is attached to a gas washing-bottle as described in the preceding experiment. The tube is dipped beneath the surface of the molten phosphorus under water in a beaker, and by means of gentle suction a column of the liquid some 10 cm. long is drawn into the tube. The lower end of the tube is then sealed with a small deflagrating-spoon filled with water and the tube immersed in a beaker of cold water. In a few moments the phosphorus will have solidified and, by removing the rubber tube, the stick may be forced out under water with a glass rod. By selecting tubes of different sizes it is easy to obtain rods of phosphorus of any size desired. A rubber tube should connect the gas washing-bottle with the mouth, and a pinch-cock may be advantageously employed

to close the tube after the phosphorus has been drawn up. In using a deflagrating-spoon to seal the end of the tube it is essential that a layer of water should remain in the spoon when it is withdrawn into the air, as otherwise the phosphorus may easily become ignited.

Glass tubes of different sizes; gas washing-bottle; deflagrating-spoon; P.

4. Preparation of globules of phosphorus. — In the greater number of experiments in which phosphorus is used the amount required is exceedingly small, and consequently it is necessary either to cut off small pieces of phosphorus from a large stick or to subdivide the molten phosphorus in such a manner that it may solidify in small globules. This latter operation is easily performed by allowing molten phosphorus to flow out of a jet down through a column of ice-cold water.

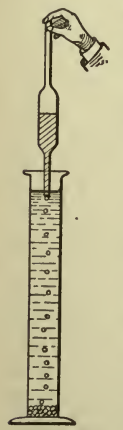

FIG. 100

A 25 cc. pipette is filled with phosphorus, as described in Ex. 4, and its tip thrust just beneath the surface of water in a tall cylinder. The cylinder is nearly filled with ice-water and then a 2 cm. layer of warm water carefully poured on top. The layer of warm water prevents the solidifying of phosphorus in the tip of the pipette. The phosphorus is then allowed to trickle slowly out of the pipette and fall through the long column of ice-cold water. During its passage the globules solidify and collect in small beads at the bottom of the cylinder. By varying the size of the tip of the pipette and regulating the flow of the phosphorus, larger or smaller globules can be obtained. The glass cylinder should be at least 40 cm. high (Fig. 100).

Minute globules, which are often advantageous for operating with phosphorus, may be obtained by melting a few grams of phosphorus under 25 cc. of water in a 100 cc. flask. When the phosphorus is melted, the flask is tightly corked and the contents well shaken. The phosphorus solidifies in very fine globules, which should be kept under water in a tightly corked bottle.

25 cc. pipette ; cylinder 40 cm. high ; P.

5. Purification of phosphorus. — Ordinary phosphorus may be purified and deprived of its dark-colored coating by melting it under a dilute solution of potassium dichromate and sulphuric acid.

The acid mixture and phosphorus should be placed in a beaker, which is in turn partly immersed in hot water in an evaporating-dish or water-bath. The phosphorus soon melts and, at the end of 15 minutes, the impurities will have been so far dissolved as to leave the phosphorus a clear, almost colorless liquid in the bottom of the beaker. The oxidizing mixture may be washed from the beaker by decantation and the purified phosphorus cast into sticks as described in Ex. 2.

Water-bath ; beaker ; yellow P ; $K_2Cr_2O_7$.

PROPERTIES

6. Spontaneous inflammability. — Phosphorus at the ordinary temperatures is energetically oxidized by the oxygen of the air and is often spontaneously ignited. A clean piece of phosphorus, if allowed to stand exposed to the air in a warm room, will oxidize and ultimately ignite. To show the spontaneous inflammability of phosphorus it is necessary to have the phosphorus finely divided. The fine globules of phosphorus prepared in Ex. 4, if dried on filter-paper and exposed to the air, will oxidize more rapidly than a large piece, and the subdivision obtained by the evaporation of a

solution of phosphorus in carbon disulphide leaves a residue of phosphorus so finely subdivided as to ignite immediately.

Phosphorus is very easily soluble in carbon disulphide, and a solution may be prepared by dissolving a 7 mm. piece of dry phosphorus in 5 cc. of carbon disulphide in a test-tube. A piece of filter-paper dipped in the solution and exposed to the air will soon take fire; for as the carbon disulphide evaporates, the dissolved phosphorus is deposited all over the fibre of the paper, and in this finely divided condition is rapidly oxidized in the air. Owing to the non-volatile nature of the phosphorus pentoxide formed, the paper itself is not burned, but only partially charred.

As the paper is not ignited under these circumstances, a letter or design may be drawn on the paper with a camel's-hair brush dipped in the solution of phosphorus. On evaporation of the carbon disulphide the phosphorus is ignited and chars the design on the paper.

The luminosity of the oxidizing phosphorus is well shown in a darkened room by drawing a design with the liquid on a board. As the carbon disulphide evaporates and the phosphorus is oxidized, the design appears in lines of fire.

Camel's-hair brush; CS_2 solution of P.

7. Spontaneous combustion effected by powdered charcoal. — A piece of well-dried phosphorus, when placed on a filter-paper and covered with powdered charcoal, is ignited. The action requires some little time, but owing to the gases condensed in the charcoal as well as its non-conductivity for heat, the temperature within the mass ultimately reaches the ignition point of phosphorus.

8. Extinguishing a flame of burning phosphorus. — Burning phosphorus is difficult to extinguish by the addition of water.

A piece of clean, dry phosphorus is placed in a small tin saucer (the cover to a baking-powder can) or iron sand-bath, and ignited. When the phosphorus is burning well, a few centimeters of water are added from a test-tube, and it will be seen that the phosphorus, melted by the heat, will partially float and continue to burn and sputter in the dish. A similar piece of phosphorus placed on an iron plate and ignited is, however, readily extinguished by covering it with sand. In experimenting with phosphorus it is advisable to have a beaker full of sand within easy reach. After the phosphorus is extinguished some care is necessary to remove the sand and extinguished phosphorus without accident, and hence it is often advisable to allow small pieces of phosphorus to "burn out," exercising care that no damage is done.

Tin saucer; iron plate; sand; gauntlets; P.

9. **Combustion on cotton.** — Phosphorus pentoxide, the product of the combustion of phosphorus in air, is volatilized with difficulty, and hence forms a protective coating to combustible material, when phosphorus is burned in contact with it. This was seen in Ex. 6, where the coating of phosphorus pentoxide prevented the combustion of filter-paper.

The experiment may be carried out on a larger scale by placing an 8 mm. piece of dry phosphorus in a little depression made in a large wad of cotton batting. On igniting the phosphorus it burns quietly, and the cotton fibres in the immediate vicinity of the burning phosphorus become somewhat charred. It will be found, however, on examining the piece of cotton, after the complete combustion of the phosphorus, that the flame has not been communicated to the fibre.

Wad of cotton batting; 8 mm. piece of dry P.

10. Combustion under water. — When a stream of oxygen is conducted through melted phosphorus, the phosphorus burns, combining with the oxygen even under water.

A few grams of phosphorus are melted under water in a large test-tube, which in turn is half immersed in a beaker of water warmed to 80°. A gentle stream of oxygen is conducted through a piece of clay pipe-stem, which is inserted in the test-tube (Fig. 101). As the oxygen comes in contact with the melted phosphorus, it burns brilliantly, forming phosphorus pentoxide, which unites with the water above the phosphorus to form phosphoric acid. A portion of the phosphorus becomes converted by the heat of the combustion into the red modification, which remains suspended in the liquid. It will be found that when the initial temperature of the phosphorus and water is as low as 60°, combustion will take place, though, owing to the heat of the combustion of the phosphorus, the water is soon warmed above this temperature. The water above the phosphorus will be found to have an intensely acid reaction at the end of the combustion.

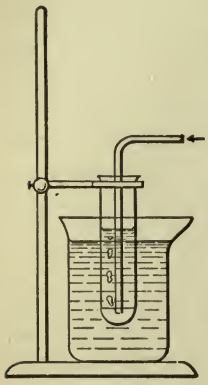

FIG. 101

$$P_4 + 5\,O_2 = 2\,P_2O_5.$$

$$3\,H_2O + P_2O_5 = 2\,H_3PO_4.$$

Apparatus (Fig. 101) ; clay pipe-stem ; O supply ; P.

11. Vaporization in a current of steam. — (*a*) Phosphorus in the presence of steam is rapidly volatilized, and the reaction of this vapor on a paper moistened with silver nitrate

solution furnishes a delicate test for the presence of free phosphorus.

A phosphorus match head is covered with 20 cc. of boiling water in a 500 cc. flask. After a few moments the water is poured out, leaving the match head in the flask. A piece of filter-paper moistened with silver nitrate solution is fastened to a wire in a cork and lowered into the flask. In the course of a few minutes the paper becomes brownish in color and finally black from the formation of silver phosphide.

A paper moistened with lead acetate solution, suspended in the flask at the same time, proves that the blackening cannot be due to the formation of hydrogen sulphide, as the lead acetate paper remains unchanged.

500 cc. flask; solutions of $AgNO_3$, $Pb(C_2H_3O_2)_2$.

(*b*) The phosphorescence of a current of steam containing phosphorus vapor is readily obtained by heating the head of a phosphorus match with 20 cc. of water in a 100 cc. flask provided with a cork carrying a short piece of glass tubing (6 mm. internal diameter). On bringing the water to a boil steam will escape from the glass tube, and in a darkened room the jet will be found to be feebly luminous from the oxidation of the phosphorus vapor.

12. Phosphorus colors the hydrogen flame green. — One of the most delicate tests for the presence of free phosphorus is based on the fact that phosphorus tints the colorless hydrogen flame green.

A hydrogen generator is constructed by inserting a straight glass tube 7 mm. in diameter through a two-holed rubber stopper in a small Erlenmeyer flask (Fig. 102). The second hole carries a glass tube so bent as to form a jet, and a platinum tip such as that described in Ex. 5, p. 183, should

be inserted in the glass tube. A few pieces of zinc are placed in the bottom of the flask and covered with water. The wide glass tube should extend to within 5 mm. of the bottom of the flask, and serves to introduce sufficient sulphuric acid to start the generation of hydrogen. After the addition of acid the flask should be shaken, and, as soon as all air is driven out, the hydrogen is lighted at the platinum tip. A phosphorus match head is softened in hot water for a few moments, and then dropped through the wide glass tube into the generator. The hydrogen flame, which was colorless, is soon colored green from the phosphorus of the match head.

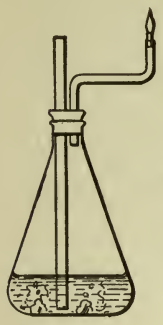

FIG. 102

150 cc. Erlenmeyer flask; glass tube (7 mm. diameter); platinum jet.

13. Action of fuming nitric acid. — At times fuming nitric acid oxidizes phosphorus to phosphoric acid with almost explosive violence. The commercial method of preparing phosphoric acid depends on this reaction, and its regulation is therefore a matter of considerable importance.

A crucible one-third filled with fuming nitric acid is imbedded in a layer of sand at the bottom of a tall glass beaker or cylinder (Fig. 103), which is placed in the hood. A 7 mm. piece of dried phosphorus impaled on the end of a long iron wire, bent in such a manner as to be easily thrust into the cylinder, is brought in contact with the fuming nitric acid in the crucible. After a few moments the phosphorus bursts into flame. When

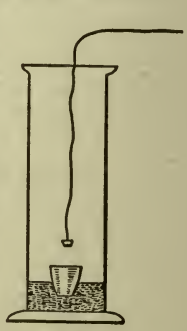

FIG. 103

the combustion is completed, the crucible may be carefully removed with the gloved hand, and the contents diluted with water. The liquid contains phosphoric acid.

Crucible ; cylinder with sand ; iron wire ; gauntlets ; fuming HNO_3 ; yellow phosphorus.

14. Action on potassium chlorate. — Phosphorus and potassium chlorate form a violently explosive mixture, which must be handled on a very small scale only.

A very intimate mixture of phosphorus and potassium chlorate is obtained by allowing 1 or 2 drops of a strong solution of phosphorus in carbon disulphide to fall from a dry test-tube upon a very small heap of finely powdered potassium chlorate placed on a brick or a piece of asbestos paper. The carbon disulphide soon evaporates, leaving a fine deposit of phosphorus all through the finely powdered potassium chlorate. As soon as the phosphorus ignites, the combustion proceeds through the whole mass with a very sharp explosion.

Powdered $KClO_3$; CS_2 solution of P.

15. Effect of pure oxygen on the glowing of phosphorus. — A stick of phosphorus, suspended by a thread, glows in a darkened room, but if lowered into a jar of oxygen, the glowing ceases.

The experiment may be varied by placing the phosphorus in a cylinder containing air, where it glows as usual. On conducting oxygen into the cylinder the glowing ceases.

Cylinder of O ; stick of P.

16. Glowing in oxygen under diminished pressure. — While phosphorus will not glow in pure oxygen at atmospheric pressure, if the oxygen is partially removed from the vessel, the phosphorus will glow in the rarefied gas.

A stick of phosphorus is lowered into a tall, narrow cylinder of oxygen fitted with a one-holed rubber stopper carrying a glass elbow. The elbow is connected with a filter-pump. The phosphorus will not glow in the cylinder of oxygen, but as soon as the filter-pump is started the glowing begins. On allowing oxygen to enter again the glow ceases. Advantageous use may here be made of a three-way stop-cock, one arm of which is inserted in the rubber stopper, the other arm connected with an oxygen supply, and the stem connected with the filter-pump. The three-way cock may be so turned as to connect with the filter-pump to produce the desired diminished pressure. By turning the cock again oxygen may be readmitted.

Tall, narrow cylinder; filter-pump; three-way cock; O supply; stick of P.

17. Action of vapors on the glowing of phosphorus. — (*a*) A stick of phosphorus placed in a glass cylinder and exposed to the air glows in a darkened room. On allowing 2 or 3 drops of ether to fall into the cylinder the glowing immediately ceases. On withdrawing the phosphorus into the air the glowing reappears. Turpentine or carbon disulphide instead of ether may be used with similar effect.

Stick phosphorus; ether; CS_2; turpentine.

(*b*) The fact that phosphorus will not glow in oxygen containing traces of certain other vapors is well shown by lowering into a jar of oxygen a piece of filter-paper on which a few drops of a solution of phosphorus in carbon disulphide have been placed. A cardboard cover should be laid over the mouth of the jar. A similar piece of paper when held in the air is ignited in a few moments, while that in the atmosphere of oxygen is not. On withdrawing the paper into the air it almost instantly catches fire.

Jar of O; cardboard cover; CS_2 solution of P.

18. Reduction of metallic salt solutions. — Phosphorus is a strong reducing agent, and precipitates many metals from their solutions.

(*a*) *Cupric sulphate.* A stick of clean phosphorus is immersed in a solution of cupric sulphate which is strong enough to appear decidedly blue. After a few minutes the phosphorus will become covered with a coating which consists of copper mixed with some copper phosphide. If the phosphorus is allowed to remain in the solution till the next exercise, all of the copper will be removed from the solution, and the clear colorless solution will give no test for copper when ammonium hydroxide is added to it.

Concentrated $CuSO_4$ solution ; stick of P.

(*b*) *Silver nitrate.* A piece of clean phosphorus, when immersed in a solution of silver nitrate, becomes covered with a metallic, crystalline coating of a mixture of silver and silver phosphide.

$AgNO_3$ solution ; stick of P.

(*c*) *Gold chloride.* A piece of clean phosphorus immersed in a rather strong solution of gold chloride is immediately coated with a deposit of gold, which, owing to its fine subdivision, appears black.

$AuCl_3$ solution ; stick of P.

AMORPHOUS PHOSPHORUS (RED PHOSPHORUS)

FORMATION AND PREPARATION

19. By the combustion of ordinary phosphorus. — Ordinary yellow phosphorus, when burned in the air, is partially converted into the allotropic form known as red phosphorus. In Ex. 4, p. 182, where phosphorus is burned in a con-

fined volume of air, considerable quantities of red phosphorus will be found remaining in the crucible lid.

20. By the action of light on yellow phosphorus. — Pure yellow phosphorus always turns dark on exposure to the light, hence it is recommended to keep the purified phosphorus in a dark place.

The change in color undergone by exposure to the light is very markedly shown by exposing to the sunlight for a few hours a tightly corked test-tube containing a stick of pure phosphorus, which is covered with water. In the course of a day of bright sunlight the phosphorus will have materially reddened, as may be seen by comparing the exposed piece with a fresh piece of pure phosphorus. The color change is still more noticeable if the lower half of the test-tube is wrapped with black paper which cuts off the light from the lower half of the stick of phosphorus. If such a piece is exposed to the sunlight, the upper half will turn a dark red, while the lower part remains unchanged.

Black paper; freshly prepared stick P.

21. By the action of iodine. — A 5 mm. piece of well-dried yellow phosphorus is heated to boiling in a test-tube. Very little, if any, change in color will be obtained. On adding a minute crystal of iodine and reheating, the whole mass becomes converted to red phosphorus.

Yellow P; I.

PROPERTIES

22. Conversion to yellow phosphorus by heat. — Red phosphorus, when heated at a high temperature, becomes converted to yellow phosphorus.

An 8 cm. length of small glass tubing is sealed at one end and a quantity of red phosphorus introduced into the tube.

On heating the phosphorus a gas escapes, which is spontaneously inflammable, the impure hydrogen phosphide resulting from the decomposition of a small amount of phosphorous acid in the red phosphorus. As the heat is increased, yellow phosphorus is formed and condenses in oily drops in the upper part of the tube.

Red phosphorus contains an appreciable quantity of phosphorous acid, and if water is allowed to pass through it on a filter-paper the filtrate will be decidedly acid to litmus.

8 cm. length small glass tubing; red P; litmus.

23. Difference in the ignition point of the two modifications of phosphorus. — The ignition point of red phosphorus is very much higher than that of yellow phosphorus. This difference may be shown by placing a small quantity of yellow phosphorus on the end of an iron bar about 15 cm. long. A small heap of red phosphorus is placed on the other end. The bar is then suspended on the ring of a retort stand and heated in the middle with a Bunsen burner. After a few moments the yellow phosphorus will ignite, but it will require considerable time and a much higher heat to cause the ignition of the red phosphorus.

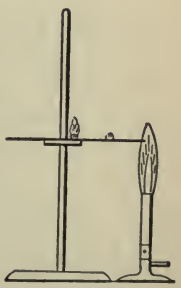

FIG. 104

The experiment may be varied somewhat by placing the yellow phosphorus at a greater distance from the flame than the red. For this purpose a strip of brass, some 20 cm. long and 2.5 cm. wide, is clamped at one end in a horizontal position (Fig. 104). A small heap of red phosphorus is placed at a point about 4 cm. from the free end and a small piece of yellow phosphorus is placed at a distance of 10 cm. from the red. The end of the brass is then heated with a Bunsen

burner. In a few moments sufficient heat will have been conducted along the strip of brass to ignite the yellow phosphorus, while the red phosphorus, which is nearer the flame, remains unchanged. On increasing the heat somewhat the red phosphorus will finally be ignited.

Iron bar (15 cm. long) ; brass strip 20 cm. by 2.5 cm. ; red P ; yellow P.

24. Combustion in air. — That the product obtained by burning red phosphorus in the air is the same as that obtained by burning yellow phosphorus, is an indication of the chemical identity of these two forms of the element.

A small quantity of red phosphorus is placed in a crucible on a plate and covered with a bell-jar. On igniting the phosphorus the combustion proceeds as with yellow phosphorus, and the bell-jar is filled with dense white fumes of phosphorus pentoxide. The oxide falls as a white powder on the plate and possesses all the properties of the product obtained by burning yellow phosphorus under the same conditions.

$$P_4 + 5\ O_2 = 2\ P_2O_5.$$

Crucible ; plate ; bell-jar ; red P.

25. Combustion with potassium chlorate. — (*a*) A mixture of red phosphorus and potassium chlorate explodes with heat or concussion.

A piece of white paper 20 mm. square is folded in the form used by druggists to dispense powders. A pinch of finely powdered potassium chlorate and about one-third the volume of red phosphorus are placed in the paper, which is very carefully folded in the original creases, care being taken to exert no pressure. The paper is then placed on an anvil and struck with a hammer in a gloved hand. The report is quite sharp.

The union between red phosphorus and potassium chlorate may be brought about by placing a small pinch of each in an unglazed mortar. By rubbing the ingredients with a pestle in the gloved hand the mixture may be exploded.

Folded paper; mortar and pestle; hammer and anvil; gauntlets; red P; powdered $KClO_3$.

(*b*) The igniting surface of a safety match-box contains red phosphorus.

A stick of potassium chlorate is prepared by carefully melting the dry, powdered salt in a small test-tube. After the contents of the tube have become liquid it is allowed to cool, and the glass is broken off, leaving a stick of solid potassium chlorate. Such a stick when rubbed on the igniting surface of a safety match-box produces a flash of fire. If the operation is conducted over a Bunsen burner through which gas is issuing, the spark produced by the friction will ignite the gas.

Stick of $KClO_3$; safety match-box.

HYDROGEN PHOSPHIDE

ORMATION AND PREPARATION

26. By the action of hydrogen on red phosphorus. — Hydrogen combines with red phosphorus directly to form hydrogen phosphide.

Dry hydrogen is conducted over red phosphorus in a 20 cm. length of combustion tubing fitted with a cork at each end, one carrying a glass tube conducting the hydrogen, the other a delivery-tube. Hydrogen is first passed through the system until all air is expelled and then the red phosphorus gently heated. Soon sufficient hydrogen phosphide is formed to render the gas collected in cylinders capable of ignition

by fuming nitric acid. If the end of the delivery-tube is allowed to dip in concentrated nitric acid, the gas becomes ignited as it comes in contact with the air.

$$P_4 + 6\,H_2 = 4\,PH_3.$$

20 cm. length of combustion tubing; H generator; red P; fuming HNO_3.

27. From phosphorus and alcoholic potash. — Phosphorus acts on potassium hydroxide, forming potassium hypophosphite and hydrogen phosphide. Certain by-products are formed in the reaction which impart to the hydrogen phosphide evolved the property of spontaneous combustibility on exposure to the air. This by-product (P_2H_4) is absorbed by alcohol, and accordingly, if a quantity of strong alcohol is added to the potassium hydroxide used in the reaction, the issuing gas will not inflame on exposure to the air.

A few pieces of phosphorus are placed in a 100 cc. Erlenmeyer flask fitted with a one-holed rubber stopper with a delivery-tube. The phosphorus is covered with 30 cc. of strong alcohol to which 15 cc. of concentrated potassium hydroxide solution have been added. The flask is gently warmed, and as soon as all air has been driven out the cork is inserted. The gas collected at the pneumatic trough is pure hydrogen phosphide. Several small jars should be collected for use in experiments on the properties of the pure gas.

$$P_4 + 3\,KOH + 3\,H_2O = 3\,KH_2PO_2 + PH_3.$$

100 cc. Erlenmeyer flask; small cylinders; con. KOH solution; alcohol; P.

28. Impure hydrogen phosphide from phosphorus and sodium or potassium hydroxide. — Impure hydrogen phosphide is especially interesting owing to its spontaneous inflammability. When prepared as described in the preceding

experiment, but without the addition of alcohol, a gas is obtained consisting chiefly of hydrogen phosphide, carrying with it small quantities of a liquid hydrogen phosphide (P_2H_4) which is spontaneously inflammable on exposure to the air. Consequently it is essential, in preparing the impure gas, to replace all air in the apparatus with hydrogen, coal gas, or other oxygen-free vapor.

Two or three 5 mm. pieces of phosphorus in a 300 cc. Jena glass flask are covered with 100 cc. of concentrated sodium or potassium hydroxide solution. Hydrogen or coal gas is passed through a tube in a two-holed rubber stopper in the neck of the flask, driving the air out of the apparatus through the delivery-tube (Fig. 105). When all air has been expelled, the supply of hydrogen is cut off and the flask is gradually heated. When the temperature of the contents of the flask is near the boiling point of sodium hydroxide, the reaction begins, and a gas is soon regularly evolved and passes over into the pneumatic trough. The first bubbles of gas rising through the water in the trough consist chiefly of hydrogen and present no special appearance. As the hydrogen is expelled and the hydrogen phosphide begins to come over, each bubble, on coming in contact with the air, is seen to take fire, burning with the formation of a white smoke. If the reaction is so regulated that the individual bubbles rise through the liquid, white rings rotating in a vortex result from the combustion of the gas. The air should be as free as possible from drafts to obtain the best results.

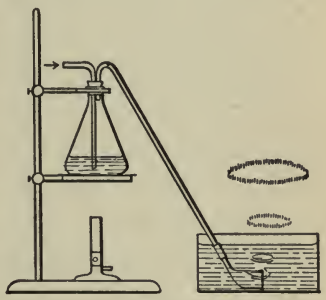

FIG. 105

To insure the formation of large bubbles the delivery-tube, or at least the end of the delivery-tube, should be of tubing of not less than 8 mm. internal diameter. On an ordinary tube the end may be enlarged by connecting to it, by means of a rubber tube, a wide tip of glass tubing. The heat must be so regulated that the gas evolution is not rapid enough to prevent the isolation of each ring.

At the end of the experiment hydrogen may be passed through the apparatus to expel the hydrogen phosphide, the current of gas being continued until the issuing bubbles are no longer ignited. The cork is then removed and water added, care being taken not to wash out any of the unused phosphorus, which should be carefully washed and transferred to the bottle containing phosphorus.

Instead of using a current of hydrogen the air may be expelled from the apparatus by adding 2 cc. of ether to the liquid in the flask before heating. In that case at the end of the reaction the flask is allowed to become perfectly cold, care being taken to have the end of the delivery-tube a considerable distance under water. As the flask cools, water is sucked back through the delivery-tube, nearly filling the flask.

Apparatus (Fig. 105); wide delivery-tube; H generator; con. NaOH; P; ether.

29. Impure hydrogen phosphide from calcium phosphide and water. — Water acts on calcium phosphide, with the liberation of hydrogen phosphide, which, as it is not pure, takes fire on exposure to the air.

A few lumps of calcium phosphide are thrown into a small cylinder containing a few cubic centimeters of water. The reaction is very rapid and large quantities of gas are liberated. As the gas comes in contact with the air at the mouth of the cylinder, it ignites and continues to burn.

The impure hydrogen phosphide thus formed may be collected if the calcium phosphide is allowed to fall into water in a small three-necked Wolff bottle. One neck of the bottle is provided with a wide, straight glass tube which dips beneath the surface of the water in the bottle. The second neck is provided with a cork and a glass elbow connected with an illuminating gas-jet, and the third neck carries a delivery-tube leading to a pneumatic trough (Fig. 106). The Wolff bottle should be about half full of water. Illuminating gas is first passed through the system until all air is removed. The current of illuminating gas is then cut off, and lumps of calcium phosphide small enough to pass through the wide tube are then allowed to fall into the bottle. Soon sufficient hydrogen phosphide will have collected in the bottle to escape at the pneumatic trough, where, as each bubble comes in contact with the air, it becomes ignited.[1]

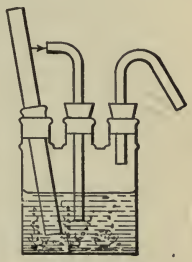

FIG. 106

$$Ca_3P_2 + 3\,H_2O = 3\,CaO + 2\,PH_3.$$

Apparatus (Fig. 106); small 3-necked Wolff bottle; wide delivery-tube; Ca_3P_2.

30. Action of calcium phosphide on water. — The rapidity of the action of calcium phosphide on water renders it difficult to obtain well-formed rings in the combustion of the hydrogen phosphide formed from the reaction. By enclosing the lump of calcium phosphide in a piece of sheet lead, according to the method described in Ex. 1, p. 39, where sodium is covered with sheet lead, the action of the water is confined to one point on the surface of the phosphide, and

[1] See note concerning delivery-tube in the preceding experiment.

hence the gas evolution is much more regular. A lump of calcium phosphide wrapped in this manner is thrown into water in a crystallizing-dish. As the bubbles of gas rise they are ignited and, in the absence of drafts, well-formed rings may be obtained.

Crystallizing-dish ; sheet lead ; Ca_3P_2.

31. Purification of hydrogen phosphide. — (*a*) *By alcohol.* The impurity in hydrogen phosphide, which causes its spontaneous combustibility, may be removed, as has been before stated, by conducting the impure gas through alcohol. The gas is conducted through alcohol in a gas washing-bottle from which all air has been expelled by a current of hydrogen or coal gas. The hydrogen phosphide issuing from the wash-bottle will be found to be so far deprived of its impurity as to be no longer spontaneously combustible.

(*b*) *By hydrochloric acid.* Concentrated hydrochloric acid acts on the impurity in hydrogen phosphide, decomposing it and forming a solid compound of phosphorus and hydrogen, which is deposited as a yellowish precipitate in the hydrochloric acid. In the apparatus used in the above experiment concentrated hydrochloric acid instead of alcohol is placed in the gas washing-bottle. After driving out the air impure hydrogen phosphide is conducted through the hydrochloric acid. The impurity is decomposed, a yellow precipitate appears, and the issuing gas is no longer spontaneously inflammable.

(*c*) *By means of cold.* Impure hydrogen phosphide when conducted through a freezing-mixture is likewise purified to such an extent that it is no longer spontaneously inflammable. The small quantity of liquid hydrogen phosphide present in the gas is condensed by the freezing-mixture.

A current of impure hydrogen phosphide is conducted through a U-tube immersed in a freezing-mixture of salt and ice. The issuing gas is not spontaneously inflammable. The condensed impurity is present in such small quantities as to be seen only after a long passage of the gas. The tube may be removed from the freezing-mixture while the gas is still passing through it and allowed to become warm in the room, when it will be observed that the issuing gas is again spontaneously inflammable.

2 gas washing-bottles; U-tube; supply of impure PH_3; ice and salt; alcohol.

PROPERTIES OF THE PURE GAS

32. Combustion in the air. — A cylinder of pure hydrogen phosphide is ignited by applying a flame. The gas burns with a bright flame, forming white clouds.

33. Ignition by heat. — A glass rod whose end has been heated in a Bunsen flame causes the ignition of a jar of hydrogen phosphide when thrust into it.

34. Ignition by fuming nitric acid. — A jar of hydrogen phosphide is instantly ignited when a glass rod which has been dipped in fuming nitric acid is thrust into it.

35. Ignition by chlorine. — Chlorine reacts with hydrogen phosphide, causing an ignition of the gas.

A 500 cc. cylinder is filled with chlorine into which is lowered the recurved jet from which issues pure hydrogen phosphide. As the gas comes in contact with the chlorine, it is ignited and burns with a greenish light.

The experiment may be varied by collecting over water 25 cc. of chlorine in a 100 cc. cylinder and allowing individual bubbles of pure hydrogen phosphide to rise inside the cylinder and come in contact with the gas. As each bubble

of gas is not instantly ignited, the cylinder must be firmly held to resist the explosion of the accumulated quantity of gas in the cylinder.

Recurved jet; 500 cc. cylinder of chlorine; 100 cc. cylinder of Cl; supply of PH_3.

36. Action with bromine vapor. — When bromine vapor is allowed to come in contact with hydrogen phosphide, a violent reaction takes place, which causes the ignition of the gas. The hydrogen phosphide used in this experiment must be pure, and not spontaneously combustible. A rod is dipped in bromine and then thrust into a small cylinder of pure hydrogen phosphide. The gas is immediately ignited.

Jar of pure PH_3; Br.

37. Ignition by silver nitrate solution. — When a strong solution of silver nitrate is poured into a cylinder of pure hydrogen phosphide, the gas is ignited.

One or two cubic centimeters of strong silver nitrate solution are poured into a 50 cc. cylinder of pure hydrogen phosphide. The gas catches fire with an explosion, and a black deposit of silver phosphide covers the walls of the cylinder.

500 cc. jar of pure PH_3; $AgNO_3$ solution (concentrated).

PROPERTIES OF THE IMPURE GAS

38. Explosion in air. — If impure hydrogen phosphide is allowed to come in contact with a confined volume of air, the combustion is of an explosive nature.

A 100 cc. glass cylinder is filled with water and inverted in a pneumatic trough. A few bubbles of air are introduced into the cylinder, and then individual bubbles of impure hydrogen phosphide are allowed to rise and come in contact with the air. As each bubble comes to the surface, a slight

explosion results, and accordingly care should be taken that the bubbles of the hydrogen phosphide ascend in the cylinder only one at a time, and that the cylinder is firmly held in the hand.

100 cc. cylinder; supply of impure PH_3.

39. Combustion in oxygen. — (*a*) The combustion of hydrogen phosphide in pure oxygen may be effected by conducting the gas in a slow stream into an apparatus consisting of a wide glass tube carrying a two-holed cork in one end. Oxygen is conducted through one hole, and the second hole carries a doubly bent glass tube having a few drops of mercury sealing the bend. The apparatus is clamped in an upright position, and a gentle stream of oxygen is conducted into the tube. As each bubble of impure hydrogen phosphide passes the mercury and ascends into the atmosphere of oxygen, it burns with a brilliant flash.

Apparatus (Fig. 87, p. 199) with bent tube; O supply; supply of impure PH_3; Hg.

(*b*) If impure hydrogen phosphide is allowed to come in contact with a confined volume of oxygen, the union is of an explosive nature.

A stout-walled 100 cc. cylinder is one-third filled with oxygen at a metallic pneumatic trough. Impure hydrogen phosphide is conducted, one bubble at a time, under the mouth of the cylinder, and allowed to rise and come in contact with the oxygen. The combustion is very sharp, making it necessary to hold the cylinder down hard on the bottom of the pan. The slow stream of hydrogen phosphide required for this experiment is best secured from the apparatus described in Ex. 29. The greatest caution must be observed to prevent an excess of the gas from rising in the cylinder.

100 cc. cylinder; O supply; supply of impure PH_3.

(*c*) A 100 cc. cylinder is filled with oxygen, covered with a pasteboard cover, and then one-fourth filled with warm water. A few centigrams of powdered calcium phosphide are then shaken into the cylinder, and the mouth nearly closed with the cardboard cover. The liberated hydrogen phosphide coming in contact with the oxygen produces a series of brilliant flashes.

100 cc. cylinder of O ; Ca_3P_2.

PHOSPHONIUM IODIDE

40. Preparation. — The action of hydrogen phosphide on iodine produces a small quantity of phosphonium iodide, which serves to illustrate the preparation of this compound.

A current of pure hydrogen phosphide is conducted through a 60 cm. length of glass tubing 2 cm. in diameter, containing a few grams of iodine near the entrance end. The iodine is gently warmed by playing a low Bunsen flame on the tube at intervals of 10 to 15 seconds. A 25 cm. length of the tube near the open end is cooled by allowing ice-water to drop on a piece of filter-paper wrapped around it (Fig. 107). If the current of hydrogen phosphide is rapid, a deposit of transparent crystals of phosphonium iodide will appear in the cooled portion of the tube. The reaction is explained as taking place in two steps: the first in which hydrogen phosphide and iodine unite to form hydriodic acid and iodide of phosphorus; in the second the excess of hydro-

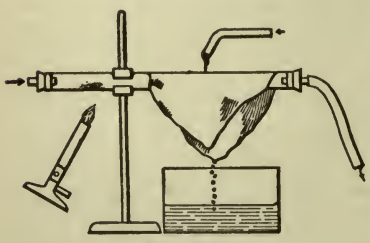

Fig. 107

gen phosphide combines with the hydriodic acid to form phosphonium iodide.

Apparatus (Fig. 107) ; 60 cm. length of combustion tubing ; supply of pure PH_3 ; I ; ice.

41. Decomposition by water. — Water decomposes phosphonium iodide with the formation of hydrogen phosphide and hydriodic acid.

Two or three grams of phosphonium iodide are placed in a small evaporating-dish on the bottom of a large beaker or battery jar, covered with a cardboard having a small hole in the centre. One or two cubic centimeters of water are poured from a test-tube on the end of a long stick, through the hole in the cardboard, upon the phosphonium iodide. At first a hissing is heard, which is followed almost immediately by an explosion.

Large beaker or battery jar ; cardboard cover ; evaporating-dish ; test-tube on stick ; PH_4I.

42. Ignition by fuming nitric acid. — Fuming nitric acid oxidizes phosphonium iodide with the liberation of iodine.

One gram of phosphonium iodide is placed on a watch-glass, and a drop of fuming nitric acid allowed to fall on it. The oxidation is accompanied by a slight noise, and iodine fumes are liberated.

Watch-glass ; PH_4I ; fuming HNO_3.

PHOSPHORUS TRICHLORIDE

43. Preparation. — By the action of dry chlorine on phosphorus, phosphorus trichloride is formed. The experiment is similar to that in which sulphur monochloride is formed (Ex. 33, p. 148).

Ten grams of yellow phosphorus are placed in a 300 cc. tubulated retort (Fig. 66, p. 148), the neck of which is

thrust through a rubber stopper deep into a filter-flask immersed in ice-water. A glass elbow extends through a cork in the tubulature, and is so arranged that it can be readily raised or lowered. Carbon dioxide is first passed through the apparatus and then a current of dry chlorine introduced. The phosphorus should be melted and sufficiently heated to have an excess of phosphorus vapor rather than of chlorine, as otherwise the excess of chlorine would tend to form solid phosphorus pentachloride instead of the liquid trichloride. The tube conducting the chlorine into the retort is depressed until it nearly touches the surface of the melted phosphorus. The neck of the retort should be tightly inserted in the neck of the filter-flask and a rubber tube should conduct the issuing gas from the side tube to a flue or some suitable absorption apparatus filled with concentrated sodium hydroxide solution. As the chlorine comes in contact with the phosphorus vapor, it ignites and burns with a feeble greenish flame. By raising the chlorine tube an excess of chlorine may be introduced in the upper part of the apparatus, when it will be seen that the neck of the retort becomes coated with crystals of phosphorus pentachloride. In order to avoid the excess of chlorine it is best to stop the operation before all the phosphorus is combined, finally cooling the apparatus in a current of carbon dioxide.

$$P_4 + 6\ Cl_2 = 4\ PCl_3.$$

Apparatus (Fig. 66, p. 148); 300 cc. retort (tubulated); filter-flask; CO_2 generator; Cl supply; ice-water; yellow P.

44. Decomposition by water. — Phosphorus trichloride is a colorless liquid somewhat heavier than water and not immediately miscible with it.

Three cubic centimeters of the chloride are placed in a test-tube and covered with 18 cc. of water. The phosphorus

trichloride remains in the bottom of the tube and does not mix with the water. On warming the tube with the hand, the water and the trichloride soon react, liberating bubbles of hydrochloric acid gas, which are absorbed before they reach the top of the liquid. By immersing the test-tube in ice-water the reaction can be entirely stopped. Decomposition again ensues on warming. If the decomposition is allowed to continue, the liquid soon becomes warm, and ultimately the phosphorus trichloride is completely converted to phosphorous and hydrochloric acids. The presence of phosphorous acid may be established by adding a few drops of gold chloride solution, which will be reduced on warming.

$$PCl_3 + 3\ H_2O = H_3PO_3 + 3\ HCl.$$

PCl_3; ice-water; $AuCl_3$ solution.

PHOSPHORUS PENTACHLORIDE

PREPARATION

45. By the combustion of phosphorus in chlorine. — A small piece of phosphorus is lowered into a liter flask filled with dry chlorine and provided with a cork carrying a calcium chloride tube. The phosphorus takes fire and burns in the excess of chlorine to form phosphorus pentachloride, which will remain as a white powder on the walls of the flask (Fig. 108). In order to insure an excess of chlorine a piece of phosphorus having a diameter of not more than 2 mm. must be used.

$$P_4 + 10\ Cl_2 = 4\ PCl_5.$$

Deflagrating-spoon; liter flask of dry Cl; cork with $CaCl_2$ tube; 2 mm. piece of yellow P.

FIG. 108

46. By the action of chlorine on phosphorus trichloride. — As was seen in Ex. 43 an excess of chlorine, when that element is acting on phosphorus, produces the pentachloride rather than the trichloride of phosphorus. Accordingly it is only necessary in preparing the pentachloride to act on the trichloride with an excess of chlorine.

The union of chlorine and phosphorus trichloride to form the solid phosphorus pentachloride may be effected by pouring 3 cc. of phosphorus trichloride into a liter flask filled with dry chlorine. A cork carrying a tube filled with calcium chloride (Fig. 108) is immediately inserted in the neck of the flask, which is then allowed to stand. After a few minutes the color will nearly all disappear from the flask, and the phosphorus pentachloride will remain as a solid mass on the bottom and walls.

$$PCl_3 + Cl_2 = PCl_5.$$

Cork and $CaCl_2$ tube; liter flask of dry Cl; 3 cc. PCl_3.

PROPERTIES

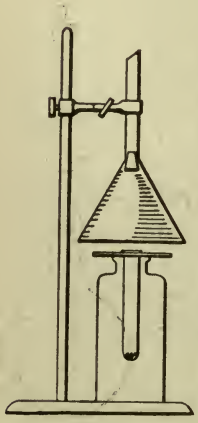

Fig. 109

47. Sublimation. — Phosphorus pentachloride sublimes without melting. A small quantity of the powder is heated in a test-tube, which is loosely corked. A sublimate soon appears on the upper part of the tube.

48. Decomposition of phosphorus pentachloride by water. — If a small quantity of water is allowed to drop on phosphorus pentachloride, the decomposition is very violent, resulting in the formation of the oxychloride.

The apparatus shown in Fig. 109 con-

sists of a wide-mouthed bottle with a cardboard cover in which an opening is made large enough to admit a test-tube. A few grams of the pentachloride are placed in the test-tube and 1 drop of water from a long bent glass tube is allowed to fall upon it. To prevent particles of the material from flying about, a large funnel having a cork in its stem is suspended mouth downwards over the test-tube. As more water is added, the conversion to phosphoric and hydrochloric acids becomes complete, the liquid remaining in the test-tube consisting of mixture of these two acids.

Phosphorus pentachloride, when allowed to drop into a large mass of water, hisses and undergoes decomposition, resulting in the formation of phosphoric and hydrochloric acids.

Apparatus (Fig. 109); PCl_5.

PHOSPHORUS BROMIDES

49. Preparation of phosphorus tribromide by the union of phosphorus and bromine. — (*a*) Phosphorus and bromine unite with explosive violence, and while no quantity of phosphorus tribromide can be prepared by this method, the interaction of the two elements may be shown by placing 1 cc. of bromine in the test-tube of the apparatus (Fig. 109) and introducing a 2 mm. piece of yellow phosphorus by means of a long stick. The phosphorus is immediately ignited, and as a rule blown out of the tube into the funnel. Care must be taken to avoid danger from the burning phosphorus, and the hands should be protected with gloves.

$$P_4 + 6\ Br_2 = 4\ PBr_3.$$

Apparatus (Fig. 109); long stick; Br; yellow P.

(*b*) The explosive violence of the reaction between phosphorus and bromine may be considerably modified by adding the bromine to phosphorus under water.

A 3 mm. piece of yellow phosphorus is placed in the test-tube of the apparatus used in the preceding experiment and covered with 10 cc. of water. Five drops of bromine are poured from a test-tube on the end of a stick upon the phosphorus. The reaction is very vigorous, and a flame is seen under the water as the two elements unite. In case the bromine is not in excess, the presence of phosphorous acid in the solution may be shown by testing with gold chloride solution.

(*c*) Phosphorus, when lowered into bromine vapor, ignites of itself and forms phosphorus tribromide, which, in excess of bromine, becomes converted to phosphorus pentabromide.

A liter flask is filled with bromine vapor by introducing a few drops of bromine and then warming the flask to expel the air. A 3 mm. piece of well-dried phosphorus is placed in a deflagrating-spoon attached to a cork and then lowered into the bromine vapor. After a few moments the phosphorus catches fire of itself and burns with a yellowish flame.

Liter flask; deflagrating-spoon; Br; P.

PHOSPHOROUS ACID

PREPARATION

50. By the slow oxidation of phosphorus. — The energetic oxidation of phosphorus produces phosphorus pentoxide, which, in the presence of water, forms phosphoric acid. If phosphorus is allowed to oxidize slowly in the air, as in Ex. 32, p. 33, in the preparation of ozone, the higher state of oxidation is not reached and considerable quantities of phosphorous acid are formed. The water remaining in the flask after the preparation of ozone, when tested with gold chloride solution, shows the presence of phosphorous acid. The ozone apparatus should be arranged and allowed to

stand, if possible, over night, when a strong test for phosphorous acid may be obtained.

$$P_4 + 3\ O_2 = 2\ P_2O_3. \quad [?]$$

$$3\ H_2O + P_2O_3 = 2\ H_3PO_3. \quad [?]$$

Ozone apparatus (Fig. 14, p. 34); $AuCl_3$; P.

51. By the action of phosphorus trichloride on water. — Water reacts energetically with phosphorus trichloride, forming phosphorous acid and hydrochloric acid. The hydrochloric acid liberated is to a large extent absorbed by the water used in the operation, though, by boiling, the excess of gas may be driven off, leaving a solution containing phosphorous and hydrochloric acids.

A few cubic centimeters of phosphorus trichloride are allowed to fall from a dropping-funnel into 20 cc. of water in a small distilling-flask. The dropping-funnel is inserted in the neck of the flask in a one-holed rubber stopper, and the side tube of the flask should be provided with a rubber tube to conduct away the hydrochloric acid fumes that are formed. As the phosphorus trichloride comes in contact with the water, a vigorous reaction takes place and large quantities of hydrochloric acid are liberated. On removing the dropping-funnel and corking the neck of the flask, the contents may be brought to a boil and the excess of hydrochloric acid driven off. The liquid will now give strong tests for phosphorous acid. A small portion of the liquid heated in a test-tube gives off first hydrochloric acid and then water. Finally, if the heating is carried to a sufficient degree, the phosphorous acid will decompose, yielding hydrogen phosphide, which may be ignited at the mouth of the test-tube.

$$PCl_3 + 3\ H_2O = H_3PO_3 + 3\ HCl.$$

Dropping-funnel; 200 cc. distilling-flask; PCl_3.

HYPOPHOSPHOROUS ACID

52. Preparation of the sodium or potassium salts by the reaction between phosphorus and the alkaline hydroxides. — See Exs. 27 and 28.

53. Reduction of sulphurous acid by sodium hypophosphite. — Hypophosphorous acid and its salts are very strong reducing agents. Sulphurous acid, itself a strong reducing agent, is reduced by solutions of sodium hypophosphite.

Strong sulphur dioxide water is treated with a solution of sodium hypophosphite and gently warmed in a test-tube. The reaction requires a little time for its completion, but soon a precipitate of sulphur is obtained.

Solution of NaH_2PO_2; SO_2 water.

PHOSPHORUS PENTOXIDE AND PHOSPHORIC ACIDS

54. Preparation of phosphorus pentoxide by combustion of phosphorus in air. — See Ex. 26, p. 29.

55. Sublimation of phosphorus pentoxide. — Phosphorus pentoxide, on heating, sublimes without melting.

A small quantity of the powder is placed in a clean, dry test-tube and heated. The oxide sublimes on the upper part of the tube, while the residue, which is combined with a small quantity of water, melts in the form of glacial phosphoric acid.

56. Evolution of heat by the action of phosphorus pentoxide on water. — When water is added to phosphorus pentoxide, sufficient heat is generated to ignite a piece of guncotton.

A small heap of the pentoxide is placed on a watch-glass and a piece of guncotton laid on it (Fig. 110). Water is allowed to drop on the oxide from the tip of a glass tube drawn out to a fine jet. As the first drop of water comes in contact with the powder, the heat generated ignites the guncotton.

FIG. 110

Watch-glass; P_2O_5; guncotton.

57. Preparation of phosphoric acids by the action of water on phosphorus pentoxide. — Phosphorus pentoxide when added to water combines with it, forming phosphoric acid.

Phosphorus pentoxide is allowed to fall in small quantities into 50 cc. of cold water in a beaker. As each particle comes in contact with the water, it hisses. Not all the oxide is dissolved, as white flocks appear in the liquid. The liquid is divided into two equal parts, one of which is brought to the boiling point; the other is filtered. On heating, the white flocks all disappear, and the solution then contains ordinary orthophosphoric acid.

The filtrate from the filtered portion contains metaphosphoric acid, which gradually becomes converted to the ortho acid. This conversion is, as might be expected, instantaneous upon heating.

$$3\,H_2O + P_2O_5 = 2\,H_3PO_4.$$

$$H_2O + P_2O_5 = 2\,HPO_3.$$

ARSENIC

ARSENIC

1. Purification of commercial arsenic. — (*a*) Commercial arsenic is generally a dull black, and in order to exhibit the metallic lustre of this element the coating must be removed. This is readily accomplished by heating the arsenic with a strong solution of potassium dichromate to which some sulphuric acid has been added. The purified material should be washed thoroughly with water and then dried with alcohol and ether.

Commercial As ; $K_2Cr_2O_7$ solution ; alcohol ; ether.

(*b*) A solution of sodium hypochlorite may be used in place of the acidulated potassium dichromate.

(*c*) The dull coating on commercial arsenic may also be removed by heating the arsenic with a small quantity of iodine in a hard-glass test-tube. The iodine first sublimes and covers the arsenic with a yellowish coating of arsenic iodide, which, on further heating, sublimes in the upper portion of the tube as a reddish sublimate of arsenic tri-iodide. The arsenic is by this operation purified, and remains as a bright metallic-looking mass in the test-tube.

But a small quantity of iodine is needed for this operation, and in a few minutes a large quantity of the impure commercial arsenic may be rendered clean and bright.

Hard-glass test-tube ; As ; I.

2. Sublimation. — Arsenic, though commonly obtained as a dull black mass, sublimes on heating to form a crystalline metallic deposit.

A few pieces of arsenic are heated in a dry hard-glass test-tube with the burner and the chimney shown in Fig. 2, p. 8. A portion of the arsenic sublimes and condenses on the upper part of the tube, that part of the sublimate nearest the source of heat having a brilliant metallic lustre and crystalline structure, while farther up the tube the deposit is of a dull gray amorphous nature.

Burner and chimney (Fig. 2, p. 8); hard-glass test-tube ; As.

ARSENIC HYDRIDE (ARSINE)

3. Preparation. — Nascent hydrogen reduces solutions containing arsenic to arsenic hydride, which is given off as a gas with the excess of hydrogen. The extremely poisonous nature of this gas renders it necessary to prepare it on a very small scale only, and to destroy it before it is allowed to escape into the room.

A hydrogen generator may be set in operation and the gas, after being conducted through a chloride of calcium tube, may be ignited at a glass jet, taking all precautions to prevent an explosion. Since the flame coloration is an essential feature of this experiment, the tube should be provided with a platinum tip or be replaced by a metallic blowpipe jet, as otherwise the naturally colorless hydrogen flame would melt the glass and become colored yellow with sodium. On adding a few drops of a solution containing arsenic the flame will be colored a bright blue by the presence of arsine in the issuing gas.

A much more satisfactory method of experimenting with this gas is shown in Fig. 111. A small wide-mouthed bottle,

containing a few grams of granulated zinc covered with water, is provided with a three-holed cork. Hydrogen is conducted from a Kipp generator through a glass elbow in one of the holes in the cork and issues through a second elbow provided with a calcium chloride tube and a jet with a platinum tip. A small thistle-tube is inserted in the third hole of the cork, its lower end being sealed with water. Hydrogen is conducted through the apparatus until all air is removed, and then sufficient hydrochloric acid is poured through the thistle-tube to produce a slow evolution of hydrogen in the bottle. On adding a few drops of a solution containing arsenic, arsine will be formed and will be conducted, along with the main hydrogen current, to the platinum jet, where it will impart a blue color to the flame.

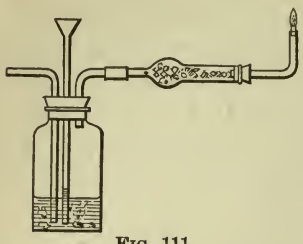

Fig. 111

H generator; Kipp generator for H; apparatus (Fig. 111); Zn; $AsCl_3$ solution.

4. Decomposition by heat. — When arsine, or its mixture with hydrogen, is conducted through a glass tube which is strongly heated, the arsine is decomposed, liberating hydrogen, and arsenic is deposited on the colder portions of the tube.

A jet is prepared by drawing a piece of 7 mm. hard-glass tubing out to a fine point. The end of the fine jet is bent at right angles, and the jet is then connected to the calcium chloride tube in the apparatus (Fig. 111). On passing hydrogen mixed with arsine through the tube and heating it strongly with a Bunsen burner, the arsenic will be deposited as a metallic mirror in the constricted portion of the tube.

If the current of arsine is not too rapid, all of the arsenic may be deposited in this manner, and consequently the color of the flame changed. For this purpose, however, it is better to use a jet of the form shown in Fig. 111, which is preferably made out of hard-glass tubing. The hydrogen should be ignited at the platinum jet and the characteristic color of the arsine flame noted. On strongly heating the tube half way between the bend and the calcium chloride tube, the arsine will be decomposed, the arsenic deposited between the flame and the bend, and the color of the arsine flame will disappear, leaving the colorless flame of hydrogen.

$$4\,AsH_3 = As_4 + 6\,H_2.$$

AsH_3 apparatus (Fig. 111); hard-glass tubing (7 mm.); Pt jet.

5. Deposition of arsenic by cooling a flame containing arsine. — If a cold porcelain dish is held in the flame of arsine burning at the jet in Fig. 111, arsenic will be deposited as a dark, metallic-appearing spot, which may be removed by the addition of a few drops of sodium hypochlorite solution, or by gently warming with nitric acid of a specific gravity 1.25. (For further consideration of arsenic spots, see under antimony, Ex. 3.)

ARSENIC SULPHIDE

6. Commercial arsenic sulphide in white fire. — Two parts of commercial arsenic sulphide (realgar, As_2S_2), when mixed with two parts of sulphur flowers and four parts of potassium nitrate, form a mixture which, on ignition, gives an intense white light.

Powdered realgar; KNO_3; S flowers.

ARSENIC TRIOXIDE

7. From the combustion of arsenic in oxygen. — Arsenic, when strongly heated in an atmosphere of oxygen, burns, forming arsenic trioxide.

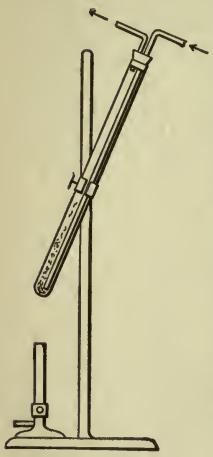

FIG. 112

The combustion of arsenic in oxygen and the sublimation of the arsenic trioxide are shown by heating a small quantity of the element in a 40 cm. glass tube 15 mm. internal diameter, sealed at one end. The tube is fitted with a two-holed cork carrying a long elbow extending nearly to the bottom of the tube and a short glass elbow connected with a flue (Fig. 112). The tube is clamped in an inclined position and a current of oxygen conducted through it. On heating the arsenic, it catches fire and burns brilliantly. Some of the arsenic is volatilized and deposited as a crystalline sublimate in the cooler portions of the tube. A considerable quantity of the arsenic trioxide formed is deposited as a white sublimate in the tube.

$$As_4 + 3\ O_2 = 2\ As_2O_3.$$

40 cm. glass tube 15 mm. diameter; cork and tubes; O supply; As.

ANTIMONY

ANTIMONY

1. Fusibility. — Antimony is easily melted with a blowpipe by directing the flame upon a small piece of the metal placed in a cavity scooped out of a piece of charcoal. The strongly heated globule, if allowed to fall upon a piece of white paper, breaks into numerous small globules, which run in all directions, each globule charring the paper over which it runs. A large pasteboard box cover may be used, or the edges of the paper may be turned up to prevent the particles of antimony from running all over the table.

White paper or box cover; blowpipe; charcoal; Sb.

2. Combustion of powdered antimony. — Pulverized antimony, when blown through a Bunsen flame, burns brilliantly, giving a white light. The finely powdered metal is placed in a glass elbow, such as is described in Ex. 18, p. 24, and blown lengthwise through the flame, either by a puff of air from the mouth or better by oxygen from a cylinder.

Glass elbow; powdered Sb; O supply.

ANTIMONY HYDRIDE (STIBINE)

3. Preparation. — The preparation of pure antimony hydride is never attempted, as hydrogen containing a small quantity of the gas gives all the characteristic tests of the hydride itself. When hydrogen is generated in solutions

containing antimony, a portion of the antimony is converted to stibine, which escapes with the hydrogen.

The apparatus used is similar to that described for arsine (Ex. 3, p. 270). Hydrogen is generated in an Erlenmeyer flask, fitted with a cork and a thistle-tube, by means of zinc and sulphuric acid. The issuing gas is dried with calcium chloride and burned at a jet. A few drops of antimony chloride solution are introduced through a funnel, and in a few moments the color of the flame will be considerably changed and a white smoke, consisting chiefly of antimony trioxide, will ascend. The reactions of stibine are very similar to those of arsine.

A cold porcelain dish held in the flame produces a dull black deposit of antimony, distinguished from the arsenic spots, which have a more metallic lustre, by being insoluble in a solution of sodium hypochlorite.

If the gas is conducted through a heated glass tube, the antimony will be deposited as a black mirror on the tube; but while arsenic is only deposited on that portion of the tube nearer the exit, antimony will be deposited on both sides of the heated portions of the tube.

The gas, when conducted through a glass elbow dipping into a beaker of silver nitrate solution, produces a black precipitate of silver antimonide.

The distinction between the reactions of the arsenic and antimony spots with sodium hypochlorite is markedly shown by holding a white dinner plate in the stibine flame and moving it in such a way that the symbol of antimony (Sb) will be written in the form of a black deposit on the plate. The plate should then be held in an arsine flame and the antimony entirely covered with a deposit of arsenic. On pouring sodium hypochlorite solution into the plate, the arsenic deposit is completely dissolved, and the antimony deposit unaffected stands out as originally deposited.

If arsine as well as stibine is present in the burning hydrogen, both elements will be deposited on a cold dish held in the flame. If, however, the dish is held in a vertical position and the spot deposited in an elongated form, the arsenic, by reason of its greater volatility, will be chiefly deposited in the upper portions of the spot. If sodium hypochlorite is allowed to act on such a deposit, the outer and upper portions will be seen to disappear, while the lower portions, consisting as they do, chiefly of antimony, remain unacted upon.

$$2\ SbH_3 = 2\ Sb + 3\ H_2.$$

$$4\ AsH_3 = As_4 + 6\ H_2.$$

Erlenmeyer flask; thistle- and delivery-tubes; evaporating-dish; plate; hard-glass tube; $SbCl_3$ sol.; NaClO sol.; AsH_3 generator.

ANTIMONY CHLORIDES

4. Combustion of antimony in chlorine. — Finely divided antimony burns brightly in chlorine, forming antimony trichloride and pentachloride.

A small quantity of antimony powder is sifted from a cheese-cloth bag into a 500 cc. cylinder filled with chlorine and placed in a strong draft. As each particle of antimony falls into the chlorine, it burns with a bright light, forming antimony trichloride, which, in the presence of an excess of chlorine, is converted to the pentachloride.

$$2\ Sb + 3\ Cl_2 = 2\ SbCl_3.$$

$$2\ Sb + 5\ Cl_2 = 2\ SbCl_5.$$

Jar of Cl; finely powdered Sb.

ANTIMONY TRISULPHIDE

5. Combustion in a current of oxygen. — Antimony trisulphide, owing to the oxidizable nature of both of its elements,

burns brilliantly in oxygen, forming antimony trioxide and sulphur dioxide.

Dry antimony trisulphide is heated in a bulb-tube in a current of oxygen. The sulphide soon takes fire and burns brilliantly. The issuing gas may be tested for the presence of sulphur dioxide by a paper dipped in potassium dichromate solution. The antimony trioxide formed is given off as a white smoke, which escapes from the tube, though a portion of the oxide is deposited as a white sublimate.

$$2\ Sb_2S_3 + 9\ O_2 = 2\ Sb_2O_3 + 6\ SO_2.$$

Bulb-tube; O supply; $K_2Cr_2O_7$ solution; Sb_2S_3.

6. Combustion in fused potassium nitrate. — Antimony sulphide is readily oxidized by being dropped into fused potassium nitrate. A small quantity of the potassium nitrate is fused in a hard-glass test-tube which is clamped in a vertical position, and antimony sulphide in the form of a fine powder is shaken into the tube. As the sulphide comes in contact with the potassium nitrate, it burns with a brilliant light. Potassium chlorate may be substituted for potassium nitrate.

Hard-glass test-tube, clamped; KNO_3; $KClO_3$; Sb_2S_3.

7. Use in Bengal fires. — Native antimony sulphide, when powdered and mixed with sulphur flowers and potassium nitrate, gives on ignition an intense white light.

One gram of finely powdered stibnite is mixed on a paper with 2 g. of flowers of sulphur and 7 g. of finely powdered potassium nitrate, avoiding all unnecessary pressure or friction. The mixed powder is placed on an asbestos paper or brick in a strong draft and ignited with a piece of touch-paper.

Powdered stibnite; S flowers; powdered KNO_3; touch-paper.

8. Preparation of antimony cinnabar. — An oxysulphide of antimony of complex composition, possessing a beautiful color and called antimony cinnabar, is obtained by slowly heating a solution of antimony chloride with a solution of sodium thiosulphate. Antimony chloride solution, to which the least possible quantity of hydrochloric acid has been added, is mixed in a flask with a solution of sodium thiosulphate and slowly heated with constant stirring to 90° on a water-bath. After a few minutes the red precipitate appears.

Water-bath ; thermometer ; $SbCl_3$ sol. ; $Na_2S_2O_3$.

BORON

1. Preparation of boron by the reduction of boric anhydride by magnesium. — Magnesium powder reduces boric anhydride at a high temperature, forming magnesium oxide and amorphous boron.

Equal volumes of finely powdered magnesium and finely pulverized boric anhydride are mixed in a hard-glass test-tube and strongly heated in a blast-lamp. The ignition tube should not be more than one-fourth filled with the mixture, and should be continually rotated while being heated. Soon a temperature will be reached at which the reaction begins, and immediately the contents of the tube glow. The tube is allowed to cool, is then broken, and the contents digested with pure dilute hydrochloric acid without warming. The excess of magnesium and the magnesium oxide are dissolved, leaving a black powder of amorphous boron, which is filtered off and allowed to dry on the paper. Preserve paper for use in the next experiment.

$$B_2O_3 + 3\,Mg = 3\,MgO + 2\,B.$$

Ignition-tube; B_2O_3 powdered; Mg powder.

2. Combustion of boron in the air. — Amorphous boron, when strewn into a Bunsen flame, burns brilliantly. A small quantity of the amorphous powder is sifted through a Bunsen flame or blown through a glass elbow (Ex. 18, p. 24) into the flame.

The filter-paper, on which the amorphous powder obtained in the preceding experiment was collected and dried, retains a considerable quantity of the powder and, when ignited, the particles of boron burn with scintillations.

Glass elbow ; filter-paper from preceding experiment ; amorphous B.

3. Preparation of boron trifluoride. — Hydrofluoric acid gas reacts with boron anhydride, forming boron trifluoride.

Hydrofluoric acid gas is generated in the presence of boric anhydride by heating a mixture of 24 g. of powdered calcium fluoride, 10 g. of powdered boric anhydride, and 20 g. of concentrated sulphuric acid in a 100 cc. Jena glass Erlenmeyer flask. A one-holed cork, carrying a glass elbow, is inserted in the neck of the flask and two dry cylinders are arranged as in Fig. 113. Provision is made for conducting the escaping gas into a flue. The flask is then gently heated, and the boron trifluoride evolved soon expels the air from the flask and the two cylinders, and fumes strongly in the air as it escapes into the flue. The corks are then withdrawn from the cylinders, which are quickly covered with glass plates.

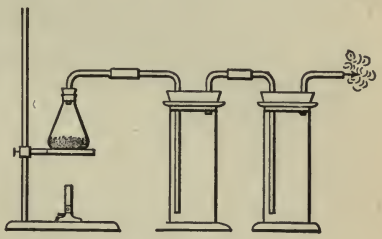

Fig. 113

$$H_3BO_3 + 3\,HF = BF_3 + 3\,H_2O.$$

Apparatus (Fig. 113) ; 100 cc. Jena glass Erlenmeyer flask ; two 200 cc. cylinders ; CaF_2 powdered ; B_2O_3 powdered.

4. Properties of boron trifluoride. — A cylinder of the gas, when opened to the air, gives off dense white fumes, which are acid to litmus paper.

A cylinder of the gas is opened with its mouth under water. The gas is absorbed, water rising in the cylinder with almost explosive violence.

5. Preparation of boric acid from borax. — Hydrochloric acid decomposes sodium diborate (borax), setting free boric acid, which, owing to its insolubility in cold water, may be easily crystallized therefrom.

Sixty grams of borax are dissolved in 240 cc. of boiling water, and concentrated hydrochloric acid is added to the alkaline solution until the reaction is decidedly acid to litmus. The beaker is then immersed in a large vessel of cold water, and the boric acid on cooling, separates out in large crystals.

$$Na_2B_4O_7 + 2\,HCl + 5\,H_2O = 2\,NaCl + 4\;H_3BO_3.$$

Large vessel of cold water ; $Na_2B_4O_7$; litmus paper (blue).

6. Dehydration of boric acid by heat. — Boric acid, when strongly heated in a crucible, loses water and fuses to a colorless anhydride which resembles melted glass.

A platinum crucible is half filled with crystallized boric acid and gently heated with a Bunsen flame. Large quantities of water vapor are evolved, and on increasing the heat the contents of the crucible are fused to a colorless liquid. If a glass rod dipped in the melted mass is withdrawn, a portion of the liquid will adhere to it and be drawn up as a fine thread, which, on cooling, solidifies. When cold, the contents of the crucible become hard as glass and are best removed by dissolving in water.

$$2\,H_3BO_3 = B_2O_3 + 3\,H_2O.$$

Pt crucible ; H_3BO_3.

7. Action of boric anhydride on water. — Twenty-five grams of powdered boric anhydride, when mixed with 30

cc. of water, combine with the water to form boric acid, with the liberation of great heat. An ether thermometer, such as was shown in Fig. 75, p. 174, when thrust into the liquid, becomes so heated that ether vapor is driven out of the top of the tube and may there be ignited. Considerable water vapor escapes into the air as a result of the heat from the reaction.

$$B_2O_3 + 3\,H_2O = 2\,H_3BO_3.$$

Ether thermometer (Fig. 75, p. 174); finely powdered B_2O_3.

8. Decomposition of sodium chloride by boric acid. — Though hydrochloric acid decomposes solutions of borates with the liberation of boric acid, boric acid will, when fused with sodium chloride, drive off the hydrochloric acid, forming sodium borate. This reaction is due to the non-volatile nature of the boric acid.

Equal volumes of fused, pulverized sodium chloride and powdered boric acid are strongly heated in a crucible. Water is first driven off from the boric acid, and finally hydrochloric acid fumes are evolved. The presence of hydrochloric acid is easily established by blue litmus paper or a rod moistened in strong ammonium hydroxide.

Porcelain crucible; fused and powdered NaCl; H_3BO_3 powdered.

9. Coloration of an alcohol flame by boric acid. — Alcohol, to which a small quantity of a solution of boric acid has been added, is poured over some asbestos in an evaporating-dish. On igniting the liquid it burns with a green flame.

A much larger flame may be obtained by boiling the mixture of alcohol and boric

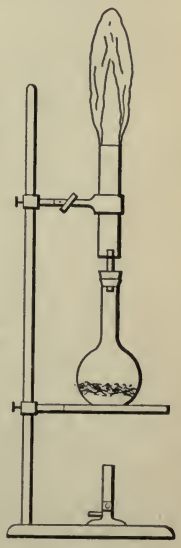

Fig. 114

acid and igniting the vapor. Three grams of finely powdered borax are mixed with 3 cc. of concentrated sulphuric acid and 20 cc. of ethyl alcohol in a 50 cc. flask, fitted with a one-holed cork carrying a short piece of glass tubing (Fig. 114). A piece of combustion tubing, 10 cm. long and 15 to 20 mm. in diameter, is vertically clamped over the small glass tube through which the vapor of alcohol is issuing. The alcohol vapor mixes with air drawn in at the lower end of the combustion tube and burns, when ignited, in a large flame at the top of the tube. The intense green color produced by the presence of boric acid is strikingly shown.

50 cc. flask; cork and short tube; 10 cm. length of combustion tubing; $Na_2B_4O_7$; alcohol.

SILICON

SILICON

1. Preparation by the reduction of silicon dioxide by magnesium powder. — Silicon dioxide, when mixed with finely powdered magnesium and strongly heated in an ignition-tube, is reduced by the magnesium to silicon. In case the magnesium is in excess, the silicon and magnesium unite to form magnesium silicide.

The reduction is well shown by heating 1 g. of clean, dry, fine, white sand with 1.5 g. of fine magnesium powder. The mixture is placed in a hard-glass test-tube, which is gradually heated by constant rotation in the flame. As the temperature increases, a point will finally be reached at which the reaction starts, and it then proceeds throughout the whole mass, which glows vividly. The black residue in the tube consists chiefly of magnesium silicide.

The preparation of a considerable quantity of crude silicon, which is used in Ex. 7, is easily accomplished by heating a mixture of 100 g. of dry, fine sand with 50 g. of magnesium power in a Hessian crucible over a blast-lamp. It is of the greatest importance that the materials should be perfectly dry. An asbestos cover and an iron plate should be laid on top of the crucible. The mixture is carefully heated, and the reduction, when once started, proceeds of itself. This product, owing to the deficiency of magnesium in the mixture, contains but a small quantity of magnesium

silicide. After cooling, the product should be treated with dilute hydrochloric acid to destroy any traces of magnesium silicide formed, washed carefully with water, and dried ready for use.

$$SiO_2 + 2\ Mg = 2\ MgO + Si.$$

Ignition-tube; Hessian crucible; asbestos; iron cover; blast-lamp; fine sand; fine Mg powder.

2. Preparation of magnesium silicide. — Silicon dioxide, reduced with an excess of magnesium, forms magnesium silicide.

Twenty grams of magnesium powder are mixed with 12 g. of fine, dry sand and heated over a Bunsen burner in an iron saucer. The mixture should be covered with a piece of asbestos paper to prevent the entrance of air. On strongly heating with the burner the reduction is completed and a product rich in magnesium silicide is obtained. The material should be powdered and placed in a dry, tightly stoppered bottle.

$$4\ Mg + SiO_2 = 2\ MgO + SiMg_2.$$

Iron saucer; asbestos paper; bottle; magnesium powder; fine, dry SiO_2.

SILICON HYDRIDE

3. Preparation of silicon hydride from magnesium silicide and hydrochloric acid. — (*a*) Magnesium silicide reacts with hydrochloric acid, with the liberation of silicon hydride, a spontaneously inflammable gas.

A small quantity of pulverized magnesium silicide is sifted into 10 cc. of concentrated hydrochloric acid in a 50 cc. cylinder. The gas liberated catches fire on exposure to the air.

The cylinder may be filled with oxygen, thus having an atmosphere of oxygen rather than air above the hydrochloric acid. The reaction on adding magnesium silicide is very sharp.

(*b*) A considerable quantity of silicon hydride may be prepared and burned at a jet by means of the apparatus, Fig. 115. A 300 cc. bottle fitted with a two-holed rubber stopper is completely filled with water and a few grams of magnesium silicide, prepared as in the preceding experiment, are placed on the bottom. A straight glass tube extends through the cork nearly to the bottom of the bottle, and is fitted by means of a cork to the tubulature of an inverted 500 cc. bell-jar. A glass elbow, inserted in the second hole of the cork, is connected by means of a rubber tube and pinch-cock with a glass jet 2 mm. internal diameter. Twenty cubic centimeters of concentrated hydrochloric acid are poured into the bell-jar, and, flowing down through the straight glass tube, come in contact with the magnesium silicide. The silicon hydride formed collects in the top of the bottle and forces the liquid up into the bell-jar. By carefully opening the pinch-cock a stream of silicon hydride, mixed with some hydrogen, escapes. On coming in contact with the air the gas spontaneously ignites, burning with a bright flame.

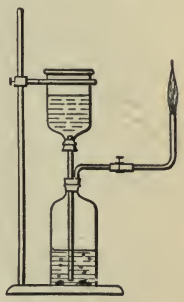

Fig. 115

An iron plate held in the upper part of the flame becomes covered with a white deposit of silicon dioxide, one of the products of combustion.

A white porcelain dish held in the lower part of the flame is covered with a brown deposit of amorphous silicon.

$$SiMg_2 + 4\,HCl = 2\,MgCl_2 + SiH_4.$$

$$SiH_4 + 2\,O_2 = SiO_2 + 2\,H_2O.$$

Apparatus (Fig. 115); 300 cc. bottle; bell-jar; tubes and pinch-cock; iron and porcelain dishes; $SiMg_2$.

(*c*) Silicon hydride mixed with varying amounts of hydrogen (which for most experiments does no harm) may easily be obtained by using as a generator a small three-necked Wolff bottle, in which the magnesium silicide is placed and covered with water. Hydrogen from the Kipp generator enters through a glass elbow, which is thrust into one neck of the bottle and extends beneath the surface of the water. A dropping-funnel in the middle neck contains concentrated hydrochloric acid, while the third neck is provided with a cork and a delivery-tube. Hydrogen is first passed through the apparatus to drive out all air, and then concentrated hydrochloric acid is allowed to fall upon the magnesium silicide. The silicon hydride generated, mixed with considerable quantities of hydrogen, passes out through the delivery-tube and ignites upon coming in contact with the air. By carefully regulating the flow of hydrogen from the Kipp generator quite pure silicon hydride may be obtained.

500 cc. three-necked Wolff bottle ; dropping-funnel ; cork and delivery-tube ; H generator.

4. Decomposition by heat. — Hydrogen containing silicon hydride or the pure gas, silicon hydride, is passed through a glass tube, which is strongly heated. A deposition of brown, amorphous silicon is obtained.

5. Combustion in the air. — A cylinder of silicon hydride is collected over water, covered with a glass plate, and placed mouth upwards on the table. On removing the plate, the gas ignites of itself and burns. Owing to the deficiency of air, a deposit of brown amorphous silicon is left on the inside of the cylinder.

6. Explosion with air or oxygen. — Air or oxygen, when allowed to enter a confined volume of silicon hydride, produces an explosion.

Twenty cubic centimeters of silicon hydride are collected in a 100 cc. cylinder, and one or two bubbles of air are allowed to rise inside the cylinder. A sharp explosion is obtained.

If oxygen is admitted to a confined volume of the hydride, the greatest care must be exercised that not more than one bubble is introduced at a time.

The conditions of this experiment may be reversed and a bubble of silicon hydride allowed to enter a confined volume of air or oxygen. The introduction of silicon hydride is best made from the apparatus of Fig. 115, p. 285, which admits of a careful regulation of the flow of the gas.

Pneumatic trough ; cylinders ; O supply ; SiH_4 supply.

SILICON TETRACHLORIDE

7. Preparation by the action of chlorine on crude silicon. — A quantity of dry, crude silicon, prepared as in Ex. 1, is placed

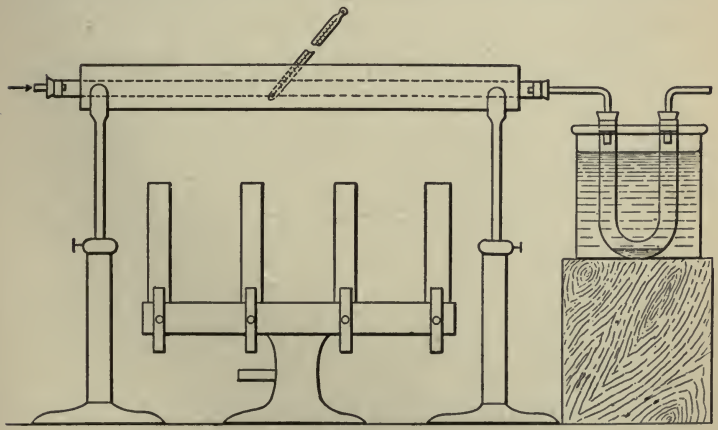

Fig. 116

in a 50 cm. length of combustion tubing, through which dry chlorine is conducted (Fig. 116). As the quantity of silicon

tetrachloride formed varies greatly with the temperature, it is necessary to heat the tube in an air-bath consisting of a tin or sheet-iron trough covered with a piece of asbestos paper. A thermometer is placed in the air-bath, which should be heated by means of a four-tube burner to 345° C. The issuing gas is conducted through a U-tube immersed in ice-water, and the silicon tetrachloride condenses to a light yellow liquid.

$$Si + 2\,Cl_2 = SiCl_4.$$

50 cm. length combustion tubing; air-bath; four-tube burner; thermometer; U-tube immersed in ice-water; Cl generator (Fig. 39, p. 81); crude silicon from Ex. 1.

8. Decomposition by moisture. — (*a*) Silicon tetrachloride fumes strongly in the air, from which it abstracts moisture. The reaction is accompanied by a liberation of hydrochloric acid and a white powder consisting of silicic acid remains. A few cubic centimeters of the liquid are poured into a glass crystallizing dish and are allowed to stand exposed to the air. A piece of moist blue litmus paper is immediately reddened when exposed to the fumes. When the reaction is complete, a white powder is left in the bottom of the dish.

Glass crystallizing dish; litmus paper (blue); $SiCl_4$.

(*b*) Silicon tetrachloride unites directly with water, forming hydrochloric acid and gelatinous silicic acid.

A few drops of the tetrachloride are allowed to fall into 2 cc. of water in a test-tube. Gelatinous silicic acid is formed and hydrochloric acid escapes.

$$SiCl_4 + 4\,H_2O = H_4SiO_4 + 4\,HCl.$$

SILICON TETRAFLUORIDE AND HYDROFLUOSILICIC ACID

9. Preparation of silicon tetrafluoride. — When hydrofluoric acid gas is generated in the presence of silicon dioxide, silicon tetrafluoride is formed.

Thirty grams of finely pulverized calcium fluoride are mixed with an equal weight of fine sand and heated with sufficient concentrated sulphuric acid to make a thin paste. The mixture is placed in a 250 cc. flask fitted with a two-holed rubber stopper. A double safety-funnel, such as is shown in Fig. 85, p. 196, containing mercury, is placed in one hole of the stopper, and the issuing gas is conducted through a glass elbow in the second hole to the bottom of a dry 400 cc. cylinder arranged as in Fig. 113, p. 279. Two cylinders are placed in series and filled with the dry gas by gently heating the flask.

$$SiO_2 + 4\,HF = SiF_4 + 2\,H_2O.$$

250 cc. flask ; two-holed rubber stopper ; safety-funnel ; two 400 cc. dry cylinders ; fine SiO_2 ; finely pulverized CaF_2 ; Hg.

10. Silicon tetrafluoride is a non-supporter of combustion. — A burning candle thrust into a jar of silicon tetrafluoride is immediately extinguished. The gas itself does not burn.

Candle on wire ; jar of SiF_4.

11. Decomposition of silicon tetrafluoride by water vapor. — Silicon tetrafluoride is decomposed by the action of water, forming hydrofluosilicic and silicic acids. A jar of silicon tetrafluoride, when opened in moist air, fumes strongly.

A glass rod moistened with water, when lowered into a jar of the gas, will become covered with a deposit of white silicic acid.

If silicon tetrafluoride is slowly conducted into the lower end of a vertically clamped piece of combustion tubing 60 cm. long, the decomposition of the gas by water-vapor may be more strikingly shown. Moisture should be introduced into the combustion tube just before use by blowing through it with the mouth. As the gas slowly rises in the tube, the walls become covered with a white deposit of silicic acid.

60 cm. length of combustion tube; jars of SiF_4; SiF_4 supply.

12. Preparation of hydrofluosilicic acid by the action of silicon tetrafluoride on water. — Silicon tetrafluoride is conducted into water, and, to prevent the stoppage of the tube by the silicic acid formed, the end should dip under a 2 cm. layer of mercury. The mercury may also be placed in a small crucible in the bottom of the cylinder. As each bubble of gas rises through the water it becomes coated with a film of silicic acid, which collects as a froth on the surface of the water.

$$3\,SiF_4 + 4\,H_2O = H_4SiO_4 + 2\,H_2SiF_6.$$

200 cc. cylinder; small porcelain crucible; SiF_4 supply; Hg.

CARBON

CARBON

1. Preparation of charcoal by heating organic material out of contact with the air. — Organic material, when heated out of contact with the air, loses a great part of its water and becomes converted to charcoal.

(*a*) A few small pieces of wood are placed in the bottom of a small Hessian crucible, covered with a layer of fine sand, and strongly heated till no more fumes are given off. The combustible nature of the gases evolved may be shown by igniting them. The crucible is allowed to become cool, the sand is shaken out, and the small pieces of charcoal removed. The great shrinkage in the wood may be shown by having a number of pieces of wood of the same size, of which only a part are heated.

(*b*) Sugar may be charred in a porcelain crucible and the carbonaceous residue exhibited.

(*c*) In the dry distillation of wood (Ex. 61, p. 324), the residue of charcoal may be removed from the tube.

Hessian crucible ; porcelain crucible ; sand ; small pieces of wood ; sugar.

2. The electrical conductivity of carbon. — The electrical conductivity of the various forms of carbon may be shown by closing an electric circuit with a piece of charcoal, a bit

of electric-light carbon, a pencil sharpened at both ends, and a piece of graphite. A fine piece of iron wire or a piece of platinum wire, such as is used in suspending Welsbach mantles, connects the two upright wires shown in Fig. 22, p. 53. Four or five cells of a bichromate battery are connected with the terminals of these wires, leaving a gap in the circuit which can be closed by the carbon conductors. On closing the circuit, the platinum wire will be heated to redness or even melted, while the iron wire, if used, will be ignited and burn in the air. The circuit between a dry battery and an electric bell may be used in place of the platinum wire and large battery.

Wires on block (Fig. 22, p. 53) ; bichromate battery ; fine Fe or Pt wire ; pieces of charcoal ; electric-light carbon rod ; lead-pencil ; graphite.

3. The absorption of hydrogen sulphide by charcoal. — (*a*) Air from a water-blast or gasometer is conducted through a gas washing-bottle one-third filled with a strong aqueous solution of hydrogen sulphide. A T-tube connects the gas washing-bottle with an 80 cm. length of combustion

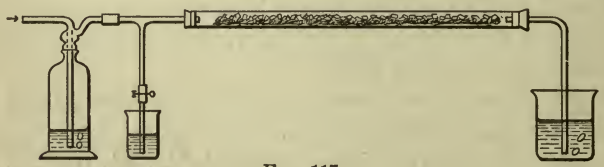

Fig. 117

tubing filled with coarsely pulverized charcoal. The stem of the T-tube is connected by means of a short piece of rubber tubing and a pinch-cock with a glass tube dipping into a dilute solution of lead acetate in a beaker (Fig. 117). The other end of the combustion tube is provided with a cork and a glass elbow dipping into a beaker containing lead acetate

solution. By opening the pinch-cock and closing the end of the combustion tube, the air containing hydrogen sulphide is caused to bubble through the lead acetate solution, producing a black precipitate. On closing the pinch-cock, the air and hydrogen sulphide proceed through the combustion-tube filled with charcoal, and then bubble through the lead acetate solution in the beaker at the end of the combustion-tube. Here it will be observed that no discoloration will take place, as all the hydrogen sulphide has been absorbed by the charcoal. The rate at which the air is conducted through the system should not be too fast to admit of counting the bubbles.

Gas washing-bottle; 80 cm. length of combustion tubing; current of air; H_2S solution; coarsely pulverized charcoal.

(*b*) Bone-black absorbs gases from their solution in water as well as from air, and if a weak solution of hydrogen sulphide is shaken with an excess of bone-black and then filtered, it will be found that the filtrate will no longer smell of the gas nor give any of its reactions.

H_2S solution; bone-black.

4. Absorptive power of carbon for coloring matter. — The use of finely divided carbon, especially in the form of bone-black, in decolorizing liquids is shown by boiling 50 cc. of litmus solution with 10 g. of bone-black. On filtering off the bone-black, the filtrate will be found to be colorless.

If double the quantity of bone-black is used and the flask is vigorously shaken, the application of heat is unnecessary.

The bone-black may be placed in a vertically clamped wide glass tube having a layer of asbestos next the one-holed cork in the lower end. If litmus solution is allowed to percolate slowly through the long layer of bone-black, the color is discharged.

40 cm. length tubing (15 mm. diameter); asbestos; bone-black; litmus solution.

5. Absorption of salts from their solutions by bone-black. — Finely divided carbon removes salts, as well as coloring matters, from solutions, as can be seen by boiling 100 cc. of a solution of lead nitrate containing .5 g. of salt to the liter with 10 g. of animal charcoal. On filtering off the liquid, the addition of a solution of hydrogen sulphide to the clear filtrate will produce no precipitate, while the original solution will yield, on the addition of hydrogen sulphide, a black precipitate of lead sulphide.

A solution of acid sulphate of quinine possessing a decidedly bitter taste may be similarly treated with animal charcoal, and afterward the filtrate will no longer taste of quinine.

.5 g. $Pb(NO_3)_2$ in 1 l. of water; bone-black; acid sulphate of quinine.

6. Combustion of graphite in oxygen. — In spite of the great fire-resisting properties of graphite, it will, when heated to a sufficiently high temperature, burn in an atmosphere of oxygen to form carbon dioxide.

A small piece of pipe-stem or fire-brick is fastened to a piece of stout wire, which is in turn fastened to a cork. A hollow should be made in the brick, by means of a file and a small piece of graphite laid in it. By directing a jet of oxygen through a glass tube, held in a small gas or candle flame, sufficient heat will be developed to ignite the graphite. When glowing strongly in the air, the graphite is lowered into a wide-necked flask filled with oxygen by displacement, where it will continue to glow and burn to carbon dioxide.

$$C + O_2 = CO_2.$$

200 cc. flask (wide-necked); fire-brick or pipe-stem; stout wire; O supply; graphite.

7. Combustion of a diamond in oxygen. — Diamond, like other forms of carbon, when heated strongly, burns in oxygen to form carbon dioxide.

A piece of pipe-stem, 1 cm. long, is hollowed a little in one end with a file and the hole plugged with a piece of asbestos paper. A copper wire is bent like a hook and the pipe-stem fitted on the end. The wire is fastened to a cork fitting a 250 cc. wide-mouthed flask filled with oxygen, and is cut at such a length that when the cork rests in the neck of the flask the bend in the wire nearly touches the bottom. The diamond[1] is placed in the hollow in the upper end of the pipe-stem and heated to incandescence with a small gas flame, through which a gentle current of oxygen is directed (Fig. 118). The table should be covered with a large piece of glazed paper, such as is used in transferring precipitates, to catch the diamond in case it should be shaken off the wire. When the diamond is strongly glowing, the gas flame may be extinguished and the stream of oxygen allowed to play upon it. The combustion is very vivid. The oxygen stream should then be cut off and the diamond allowed to cool in the air. It will be seen that the glowing almost immediately stops. On again heating the diamond and lowering it into the flask of oxygen, it will glow for some little time, and the presence of carbon dioxide in the flask may be established by adding 20 cc. of a solution of barium hydroxide and shaking the flask. A precipitate of barium carbonate will be formed.

Fig. 118

[1] Small crystals of diamond, with the longer axis approximately 2 mm. long, may be obtained of George L. English & Co., 3 and 5 West 18th St., New York City, at a cost of 25 cents per crystal. Crystals of this size and cost have been used by the writer and are large enough to give good results in this experiment.

To prevent the possible introduction of carbon dioxide into the flask from the flame used in heating the diamond, it is best to cut off the gas supply and allow the diamond to glow in the jet of pure oxygen a moment before lowering it into the flask. The flask should be filled with oxygen by displacement, in order to avoid wetting the interior.

250 cc. wide-mouthed flask ; wire and pipe-stem ; O jet ; small crystal or fragment of diamond ; $Ba(OH)_2$ sol.

8. Oxidation of carbon at low temperatures. — The oxidation of carbon at the temperature of the room is interestingly shown by electrolyzing dilute sulphuric acid in an electrolytic apparatus, using carbon electrodes (Ex. 24, p. 95). It will be found that, while there is a very vigorous evolution of hydrogen from the negative pole, there will be very little, if any, gas ascend from the positive pole. If the two electrodes are carefully filed and rubbed with emery paper before the experiment, the positive electrode will be found to be considerably corroded, while the negative electrode will appear unacted upon. The disintegration of the positive electrode is further made apparent by the presence of small particles of carbon in the liquid and on the rubber stopper holding the electrode.

Electrolytic apparatus (Fig. 46, p. 95), with carbon electrodes ; 10 per cent H_2SO_4 ; battery.

CARBON MONOXIDE

PREPARATION

9. From carbon dioxide and carbon. — Carbon dioxide, when passed over heated carbon, becomes reduced to carbon monoxide.

Coarsely pulverized charcoal is placed in a 30 cm. length of combustion-tubing, and a gentle current of dry carbon

dioxide is conducted through the tube, which is strongly heated with a four-tube burner (Fig. 119). The issuing gas is conducted through a U-tube containing soda-lime, and issues through a vertical piece of glass tubing, which serves

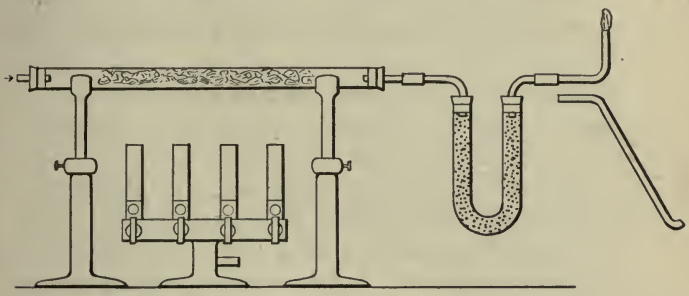

Fig. 119

as a jet. The gas issuing from the jet, when ignited, burns with a blue flame. The gas may be collected in cylinders at the pneumatic trough. A large flame is obtained by filling a liter cylinder with the gas and, after igniting it, pouring water rapidly into the cylinder.

$$CO_2 + C = 2\,CO.$$

30 cm. length combustion-tubing; 4-tube burner; liter cylinder; soda-lime; U-tube; CO_2 generator; charcoal.

10. From carbon dioxide and zinc. — Zinc dust at a low red heat effects the reduction of carbon dioxide to carbon monoxide.

A Jena glass combustion-tube is filled, for about 30 cm. of its length, with a layer of zinc dust, over which a current of carbon dioxide is passed. A cork, carrying a glass elbow which serves as a jet, is inserted in the other end of the tube. On gradually heating the tube with a four-tube burner, the reduction begins, and soon sufficient carbon monoxide

will issue from the jet to be lighted and continue to burn. At the end of the operation it will be seen that a portion of the zinc dust has been converted to white zinc oxide.

$$CO_2 + Zn = ZnO + CO.$$

Jena glass combustion-tube; CO_2 generator; 4-tube burner; Zn dust.

11. By the action of sulphuric acid on oxalic acid. — Sulphuric acid reacts with oxalic acid to form equal volumes of carbon monoxide and carbon dioxide.

Thirty grams of crystallized oxalic acid are placed in a liter flask and mixed with 100 cc. of concentrated sulphuric acid. The flask is provided with a two-holed cork carrying a long thistle-tube and a delivery-tube connected with a gas washing-bottle. The contents of the flask are gently heated, and the evolution of the gas is controlled by the application of heat. The liberated gas is conducted through a strong solution of potassium hydroxide in a gas washing-bottle, to deprive it of the greater portion of carbon dioxide. A U-tube filled with soda-lime, connected to the exit tube of the gas washing-bottle, removes the last traces of carbon dioxide, and the pure carbon monoxide may be ignited at a glass jet. The apparatus is identical with that shown in Fig. 120, save that the thermometer there shown is not used.

$$H_2C_2O_4 = CO_2 + CO + H_2O.$$

Liter flask; thistle-tube; delivery-tube; gas washing-bottle; U-tube; soda-lime; $H_2C_2O_4$; KOH.

12. By the action of sulphuric acid on potassium ferrocyanide. — The complex reaction between sulphuric acid and potassium ferrocyanide furnishes a ready means of obtaining pure carbon monoxide, and for preparing this gas in large quantities on the lecture-table this method is by far the best.

Forty-five grams of pulverized potassium ferrocyanide are mixed with 300 cc. of concentrated sulphuric acid in a 2 l. flask. The flask is closed with a three-holed rubber stopper, carrying a thistle-tube extending below the liquid in the flask, a glass elbow, and a thermometer, the bulb of which is immersed in the liquid. Care should be taken to have that portion of the thermometer reading between 150° and 175° uncovered by the cork. The issuing gas is conducted through a gas washing-bottle containing potas-

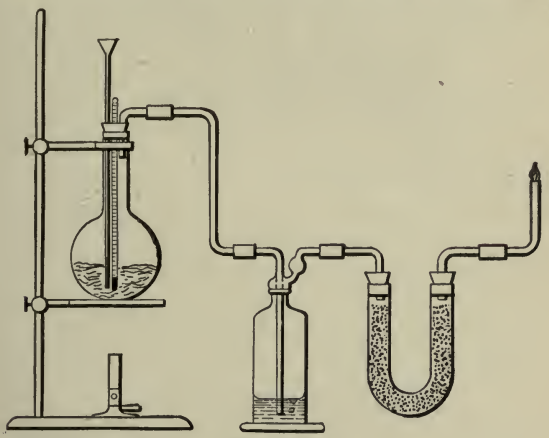

Fig. 120

sium hydroxide, followed by a U-tube containing soda-lime (Fig. 120). The flask is gradually heated and the temperature maintained at 170°, where a very regular evolution of the gas will be obtained. The pure gas issuing from the soda-lime tube may be ignited or collected in jars at the pneumatic trough, for use in any of the experiments described beyond. On removing the flame and allowing the contents of the flask to cool, the evolution of gas ceases. By heating the mixture again to 170°, even after long stand-

ing, a steady evolution of carbon monoxide is obtained. If the temperature is not allowed to rise above 175°, no difficulty in the preparation of the gas by this method will be experienced.

2 l. flask; thistle-tube; elbow; thermometer; soda-lime; U-tube; pulverized K_4FeCy_6.

13. Preparation of water gas by the action of steam on hot charcoal. — By passing steam over glowing charcoal, the water vapor becomes partially reduced by the carbon, and a mixture of hydrogen and carbon monoxide, *i.e.*, water gas, issues from the tube.

A combustion-tube is prepared in a manner similar to that described for the decomposition of nitric acid (Ex. 79, p. 226). The tube is filled with fine lumps of charcoal, which are strongly heated by means of a four-tube burner. Steam, generated in the apparatus described in Ex. 2, p. 41, is conducted through a glass tube, surrounded with a roll of asbestos paper inserted in one end of the combustion-tube, which prevents the deposition of water and subsequent fracture of the tube. If the issuing gas is collected at the pneumatic trough, it will be found to be combustible.

$$H_2O + C = CO + H_2.$$

Combustion-tube surrounded with wire gauze (Ex. 79, p. 226); asbestos paper; steam generator (Ex. 2, p. 41); 4-tube burner; charcoal.

PROPERTIES

14. Influence of moisture upon the combustion of carbon monoxide in the air. — Carbon monoxide, dried by passing through a U-tube containing pumice-stone drenched with sulphuric acid, will burn in the air; but if the jet is lowered into a cylinder containing dry air, the flame will be extinguished.

Twenty-five cubic centimeters of concentrated sulphuric

acid are poured into a 500 cc. glass-stoppered wide-mouthed specimen bottle, and the air is thoroughly dried by carefully shaking the well-stoppered bottle. A current of carbon monoxide, dried in the manner described, is allowed to burn from a recurved jet. On removing the stopper of the specimen bottle and lowering the jet into the jar, the flame will be extinguished. The gas should be passed through solid absorbents for carbon dioxide and water to prevent the flickering of the flame caused by the bubbling of the gas through a liquid. A U-tube filled with soda-lime and a similar tube containing pumice-stone and sulphuric acid will effectively purify and dry the gas.

500 cc. glass-stoppered wide-mouthed specimen bottle; recurved jet (Fig. 41, p. 85); CO supply.

15. Explosion of a mixture of carbon monoxide and oxygen. — A mixture of 2 volumes of carbon monoxide and 1 volume of oxygen explodes with considerable violence.

A 100 cc. cylinder is two-thirds filled with carbon monoxide and the remaining third with oxygen. The mouth of the cylinder should be covered with a piece of cardboard having an 8 mm. hole in the centre. The cylinder should be removed from the pneumatic trough, placed mouth upward on the table, and the flame applied at the hole in the cardboard. The explosion is quite sharp, blowing the cardboard into the air.

The explosive gaseous mixture may also be made in a round-bottomed ginger-ale bottle, as in Ex. 30, p. 67, and there exploded.

100 cc. cylinder; cardboard with hole in centre; ginger-ale bottle; CO supply; O supply.

16. Absorption by cuprous chloride. — (*a*) A hydrochloric acid solution of cuprous chloride absorbs carbon monoxide readily.

A quantity of the gas is collected in the eudiometer (Fig. 11, p. 26), and an acid solution of cuprous chloride is allowed to flow down through it. If no impurities are present, the gas will be completely absorbed.

(*b*) The absorption of this gas in cuprous chloride solution may also be shown by conducting the gas through a series of three wash-bottles, the middle one of which contains the cuprous chloride solution, the others containing water. The wash-bottles are not more than a third filled with liquid. If a current of carbon monoxide is passed through the system, the difference in the rate of bubbling in the first and last bottles will be very apparent. On heating the solution, carbon monoxide is again liberated.

Eudiometer (Fig. 11, p. 26); 3 gas washing-bottles; CO supply; Cu_2Cl_2 in HCl.

17. Action on an ammoniacal solution of silver nitrate. — Carbon monoxide reduces an alkaline solution of silver.

A gentle current of the gas, when conducted through a solution of silver nitrate, to which a slight excess of ammonium hydroxide has been added, produces a black precipitate of metallic silver.

CO supply; $AgNO_3$ solution.

18. Action on palladious chloride. — Carbon monoxide reduces solutions of palladious chloride to black metallic palladium.

A paper moistened with palladious chloride solution furnishes a delicate test for carbon monoxide, for, when held in this gas, it is immediately blackened.

CO supply; $PdCl_2$ solution.

19. Reduction of palladious chloride by a cuprous chloride solution of carbon monoxide. — The delicacy of the reaction

between carbon monoxide and palladious chloride is shown by adding 3 drops of a cuprous chloride solution of carbon monoxide to 400 cc. of water in a beaker. One drop of palladious chloride solution is added with thorough stirring. In a few moments the contents of the beaker become a deep black from the precipitated palladium.

CO dissolved in Cu_2Cl_2 solution; $PdCl_2$ solution.

20. Reduction of a solution of iodic acid. — A current of carbon monoxide, conducted into a warm solution of iodic acid, reduces the acid, forming carbon dioxide and liberating iodine.

A solution of iodic acid is gently heated in a beaker, and a current of carbon monoxide is allowed to bubble through the liquid. A filter-paper moistened with starch solution and held above the liquid is turned blue by the iodine liberated and vaporized.

CO supply; HIO_3 solution; starch solution.

CARBON DIOXIDE

PREPARATION

21. Combustion of charcoal in a confined volume of oxygen. Volumetric relation of the carbon dioxide to the oxygen consumed. — Carbon burns in oxygen to form 1 volume of carbon dioxide for every volume of oxygen used. If charcoal is burned in a confined volume of oxygen, the volume of the product will be the same as that of the oxygen consumed; hence there will be no difference in pressure in the interior of the vessel at the end of the combustion.

The apparatus used for this experiment is that shown in Fig. 68, p. 150. A 6 mm. piece of charcoal, preferably that used in blowpipe work, is placed on a platinum spoon,

ignited in the air, and thrust into the flask previously filled with oxygen. The heat of the burning carbon expands the gases and causes the mercury to rise in the open arm of the U-tube. After the operation is completed and the flask has regained its normal temperature, the level of the mercury in the arms of the U-tube will be found to be the same.

Apparatus (Fig. 68, p. 150); 700 cc. Jena glass distilling flask; U-tube; Hg; charcoal.

22. Preparation from magnesite by heat. — A thick-walled test-tube is partially filled with 5 or 10 g. of magnesite such as is used for analysis. A cork with a delivery-tube dipping into a glass cylinder containing lime-water is inserted in the mouth of the test-tube, which is then clamped in an inclined position (Fig. 121). On heating the magnesite, considerable quantities of carbon dioxide are given off and produce a marked cloudiness in the lime-water.

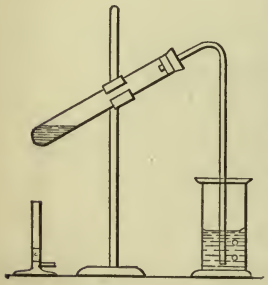

FIG. 121

$$MgCO_3 = MgO + CO_2.$$

Hard-glass test-tube; magnesite; lime-water.

23. From calcium carbonate and hydrochloric acid. — By far the most satisfactory method for obtaining carbon dioxide is to act upon calcium carbonate in the form of marble, with a mixture of equal parts of concentrated hydrochloric acid and water.

As a constant supply of carbon dioxide is very advantageous for many experiments, it is advisable to prepare a Kipp generator (Fig. 17, p. 46), filling the receptacle with small fragments of marble and using hydrochloric acid diluted as above.

The general use to which cylinders of the liquefied gas (Ex. 29, p. 307) have been applied renders it easy to procure them. They may be advantageously used for obtaining a constant stream of the gas.

Owing to its great specific gravity, carbon dioxide is almost invariably collected by displacement. Its solubility in water renders it advisable to collect the gas over mercury, in case the displacement method cannot be used.

$$CaCO_3 + 2\,HCl = CaCl_2 + H_2O + CO_2.$$

Kipp generator for CO_2; marble fragments.

24. From baking-powder. — A few grams of baking-powder are heated in a dry test-tube and the issuing gas tested with a lighted splinter. The presence of organic vapors interferes with this experiment somewhat, and the powder should be but gently heated.

Hydrochloric acid added to some baking-powder in a cylinder gives rise to an evolution of carbon dioxide.

25. Carbon dioxide a product of fermentation. — The importance of fermentation, with the liberation of carbon dioxide during the process, makes it desirable to show this operation on the lecture table.

A mixture of 50 cc. of molasses and 400 cc. of water with about one-half of a fresh, compressed yeast-cake is placed in a 500 cc. flask fitted with a cork and an elbow. A gas washing-bottle containing lime-water is connected with the elbow, and the whole apparatus allowed to stand in a warm place till the next exercise. The carbon dioxide liberated will force its way through the lime-water, rendering it turbid. An empty gas washing-bottle may be inserted between the elbow and the bottle containing lime-water, and the gas

in this cylinder may be tested with a lighted candle at the close of the experiment.

500 cc. flask; 2 gas washing-bottles; molasses; yeast-cake; lime-water.

26. Carbon dioxide in beverages. — (*a*) The presence of carbon dioxide in wine or beer may be shown by heating a portion of the liquor in a flask fitted with a cork and a bent glass tube dipping into lime-water. On the application of gentle heat, the gas is liberated, and a cloudiness is produced in the lime-water.

FIG. 122

(*b*) A glass siphon of "soda-water" (Fig. 122) is inverted, and, by pressing the valve, a large quantity of carbon dioxide is withdrawn.

A little of the liquid from a siphon is poured into the flask used above, and the carbon dioxide, given off on standing or more quickly by gentle heating, is conducted into lime-water as before.

Wine or beer; siphon of "soda-water"; lime-water.

27. Presence of carbon dioxide in air. — If a small quantity of lime-water or barium hydroxide solution is allowed to stand for several hours in a crystallizing dish in the open air, the liquid will be covered with a white film of calcium or barium carbonate.

The formation of the carbonate may be more rapidly obtained by drawing a current of air with a filter-pump through a gas washing-bottle containing either of the above solutions. In a few minutes a marked turbidity will appear in the liquid.

Filter-pump; gas washing-bottle; crystallizing dishes; lime-water; $Ba(OH)_2$ solution.

28. Carbon dioxide in expired air. — That expired air contains carbonic acid in considerable quantities is simply shown by blowing through a glass tube into 50 cc. of lime-water in a small beaker. A white precipitate of calcium carbonate is immediately formed. Continued blowing through the tube results in redissolving the calcium carbonate by the excess of carbonic acid, forming calcium acid carbonate. Heating the solution in a test-tube drives off the carbonic acid and causes the precipitate to reappear.

In case barium hydroxide is used, the precipitate will not easily be redissolved.

100 cc. beaker; lime-water; $Ba(OH)_2$ solution.

PROPERTIES

29. Preparation of solid carbon dioxide. — When liquid carbon dioxide is allowed to expand suddenly, the temperature is lowered to such an extent that a portion of the liquid solidifies.

Liquefied carbon dioxide is an article of commerce readily obtained at a very low price, being much used in preparing aërated beverages. It is ordinarily transported in steel cylinders about 1.5 m. high and 20 cm. in diameter. When standing in an upright position with the valve end uppermost, the valve may be opened and carbon dioxide gas withdrawn. If, however, the cylinder is inverted and the valve opened, the liquefied gas is forced out at the bottom in a fine stream, which immediately expands to the gaseous form, producing a great lowering of the temperature. Numerous forms of apparatus have been devised into which the stream of gas is allowed to expand with a solidification of a portion of the gas. A black flannel bag, some 20 to 30 cm. square, is an excellent substitute for apparatus of this kind, and when securely fastened to the valve-nozzle permits the collection

of considerable quantities of solid carbon dioxide. The bag is preferably made with a drawstring which may be used to fasten it securely to the valve-piece (Fig. 123).

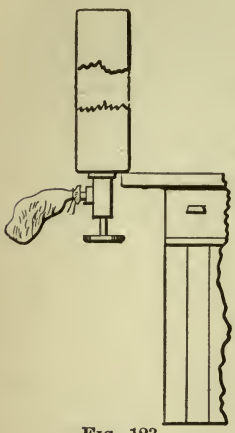

FIG. 123

As it is not infrequent in charging these cylinders with the liquefied gas that varying quantities of water are inadvertently introduced, it is important to open the valve carefully and draw off any water before fastening the bag to the valve-piece. This operation should be carried out before the lecture. As soon as all the water is forced out, the gas will begin to escape and the nozzle will become covered with frost. The bag is then attached, and, by opening the valve, a brisk stream of the liquefied gas allowed to enter. The finer particles of the solidified gas will be forced through the cloth and will fall as a white fog to the floor.

On removing the bag a considerable quantity of the solid carbon dioxide will be found as a snow-like mass which is shaken into a cardboard box. If the bag is turned inside out, it will be found to be lined with the white solid. The operation may be repeated, and the solid obtained in any quantity.

In spite of the very low temperature of the solid it remains quite a long time in the air without disappearing, and this property is especially well shown if the solid is pressed into a cake or rod, which may then be placed on a piece of felt on a plate and passed around the lecture room. A mould is made by boring a 1.5 cm. hole in a block of wood. A paper tube is fitted to the hole, and the end bent in upon

itself to make a bottom for the tube. It is then filled with the solid carbon dioxide, which is pressed down hard with a pencil or rod of wood. When the paper tube is filled, it is withdrawn, the paper unrolled, and the rod of solid carbon dioxide placed on a piece of felt or cotton-wool.

The greatest care should be exercised in handling the solid not to press it hard with the fingers; for though the rod may be laid on the hand with no sense of discomfort, the effect of pressure is to break the film of gas between the solid and the warm hand and thus cause a severe burn. So long as no pressure is applied the solid may be handled with impunity. In general, a paper scoop or a horn spatula will be found most serviceable in handling the solid.

Steel cylinder of liquefied CO_2; valve-wrench; flannel bag; cardboard box; block of wood with 1.5 cm. hole; paper tube; paper scoop; horn spatula; rod of wood or lead-pencil; felt; plate.

30. Experiments with solid carbon dioxide. — A few pieces are placed in a clean, dry cylinder and allowed to stand for a few moments. Sufficient gas will be evolved from the solid to fill the cylinder, and a lighted taper will be extinguished when thrust into the jar.

A piece of the solid is placed in the ginger-ale bottle of Ex. 30, p. 67, which is then corked with a rubber stopper. In a short time the evolution of gas within the bottle will have generated enough pressure to blow the stopper out of the neck.

A one-holed rubber stopper carrying a delivery-tube is inserted in the mouth of the bottle and the evolved gas caused to bubble through lime-water, where a turbidity will appear.

A portion of the gas escaping from the delivery-tube is also collected in a cylinder over water at the pneumatic trough.

Dry cylinder; ginger-ale bottle (Ex. 30, p. 67); rubber stopper; delivery-tube; lime-water.

31. Freezing water with solid carbon dioxide. — A small beaker is placed upon a few drops of water on a block of wood, and fragments of solid carbon dioxide are placed in it. In a very few moments the water will be frozen, and the beaker will be cemented to the wood.

Block of wood ; small beaker ; solid CO_2.

32. Freezing mercury with solid carbon dioxide. — Inasmuch as the temperature of solid carbon dioxide is $-79°$, mercury, when cooled with it, may be frozen to a hard solid.

(*a*) A few cubic centimeters of dry mercury are placed in an evaporating-dish, which is in turn placed on a wad of cotton-wool, and a centimeter layer of solid carbon dioxide is placed upon it. A wad of cotton-wool or a piece of cardboard should be placed over it and the dish allowed to stand for several minutes. On inverting the dish the mercury will be found to be a solid, which may, at times, adhere to the dish. By gentle tapping it may be dislodged, or a piece of paper may previously be placed in the dish between the mercury and the porcelain. The evaporating-dish should be inverted and the frozen mercury manipulated over a larger dish to catch any drops of the mercury as it melts. An iron wire may advantageously be frozen into the mercury to facilitate in lifting the solid.

(*b*) A much more striking method of showing the solidification of mercury consists of freezing the metal in the form of a ring, which is subsequently suspended and lowered into a jar of cold water. For this purpose a mould consisting of a block of wood in which a circular groove has been cut approximately 1 cm. deep and 1 cm. wide, the internal diameter of which is about 7 cm., is necessary (Fig. 124). The circular space is filled with a 7 mm. layer of mercury, and, after placing the mould on a large porcelain dish, the mercury is covered with a centimeter layer of solid carbon

dioxide. It is advisable to fill in the spaces around the block of wood with cotton-wool and to cover the dish with a piece of cardboard. After several minutes the mercury will be frozen solid, and by inverting the mould the ring will fall out. The mercury is then quickly raised with the horn spatula, placed upon a glass hook, and lowered into a large vessel of water which is near the freezing point. Instantly the mercury ring becomes covered with an ice ring, then melts, and the mercury falls to the bottom of the vessel. The ice ring remains on the glass hook for several moments before melting.

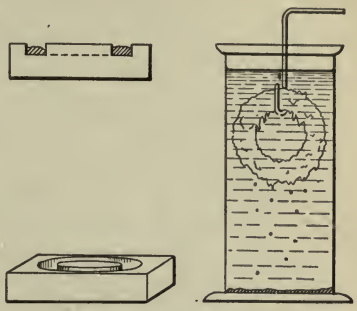

FIG. 124

While the addition of anhydrous ether lowers the temperature of the solid carbon dioxide somewhat, the mercury can be frozen without the addition of ether, provided sufficient time is allowed to complete the solidification.

Cotton-wool ; evaporating-dish ; large porcelain dish ; wooden mould; horn spatula ; glass hook ; large vessel of ice-cold water ; dry Hg ; Fe wire ; solid CO_2.

33. Specific gravity. — Carbon dioxide is much heavier than air, and is the subject of many experiments illustrating this property.

A liter beaker is supported on the arm of the lecture-balance and the system brought into equilibrium. If a current of carbon dioxide is allowed to flow from a tube held above the beaker, it will collect in the bottom, expelling the air and causing a marked increase in weight.

Instead of conducting the carbon dioxide into the beaker in a stream, a large cylinder may be filled with the gas, which may then be poured rapidly into the suspended beaker. A marked inclination of the pointer on the balance is immediately observed.

Lecture-balance; 2 l. beakers; CO_2 generator.

34. Quantitative estimation of the specific gravity of carbon dioxide. — It is possible to determine with considerable accuracy the specific gravity of carbon dioxide in a manner analogous to that described for determining the specific gravity of hydrogen (Ex. 10, p. 48).

A narrow-necked, graduated liter flask is suspended on one arm of the lecture-balance with the mouth uppermost. The system is then brought into equilibrium. A current of dry carbon dioxide is conducted through a long glass tube inserted in the neck of the flask and extending to the bottom. In a few minutes the air will have been entirely driven out, and by slowly withdrawing the tube the flask is completely filled with carbon dioxide. As the air originally in the flask has been replaced by a heavier gas, it will be necessary to add more weights to the other pan of the balance to restore the equilibrium. With a normal barometric pressure and a temperature of 15°, 0.63 g. will have to be added. A liter of air under these conditions of temperature and pressure weighs 1.225 g., hence a liter of carbon dioxide weighs 1.225 plus 0.63, which equals 1.855 g. Compared to the weight of a similar volume of air it is readily calculated that the specific gravity of carbon dioxide is 1.51.

If the barometric and thermometric conditions vary widely from those given above, it will be necessary, if the greatest accuracy is desired, to apply the usual corrections

to determine the weight of a liter of air under the observed conditions.

Lecture-balance; liter graduated flask; weights; H_2SO_4 gas washing-bottle; CO_2 generator.

35. Soap-bubbles float on carbon dioxide. — If a soap-bubble is allowed to fall into a large jar which is half full of carbon dioxide, it will settle in the jar to the level of the carbon dioxide and then float on top of this gas.

A tall, wide-mouthed jar, such as is used for large specimens in museums, is half filled with carbon dioxide. A small soap-bubble, blown on the end of a thistle-tube, is allowed to fall into the jar, where it will float on the surface of the carbon dioxide.

Thistle-tube; large jar (8 or 10 l.) half filled with CO_2; soap sol.

36. Carbon dioxide rotates a paper wheel. — The great specific gravity of carbon dioxide may also be shown by pouring a liter of the gas upon a cardboard wheel having paper cups on its periphery. As the carbon dioxide collects in the cups the wheel is caused to rotate on its axis.

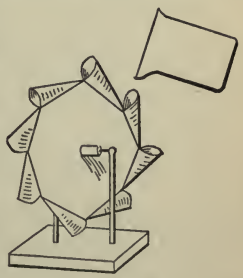

FIG. 125

A piece of stout cardboard is cut into a circle 20 cm. in diameter, ten or twelve small paper cups are pasted on the rim, and a long needle is thrust through the centre of the wheel (Fig. 125). Two stout copper wires fastened to a block of wood may be so bent as to form the bearings on which the needle is set. In balancing the wheel it will probably be necessary to add a bit of wax at different points before securing perfect equi-

librium. When once arranged, the wheel will rotate quite rapidly if a liter of carbon dioxide is poured into the cups.

Cardboard wheel; liter cylinder of CO_2.

37. Carbon dioxide may be siphoned. — By reason of its great specific gravity, carbon dioxide may be siphoned from one vessel to another.

A 2 l. cylinder is filled with carbon dioxide and its presence established by lowering a lighted candle into it. An empty cylinder of the same size is likewise tested with the candle and the absence of carbon dioxide shown. A glass tube, with an internal diameter of not less than 7 mm., bent in the form of a siphon, is inserted in the jar of carbon dioxide, which should be placed on a box or other support above the table. The siphon is started by gentle suction with the mouth on the longer arm, and, when the gas has filled the tube, the lower end is thrust into the empty cylinder. However, as the upper cylinder is fixed, it is better to arrange to have the lower cylinder brought up from beneath. The presence of the gas in the longer arm of the siphon will be known by the taste. After a few minutes the gas will all have left the upper cylinder, and a candle will continue to burn when lowered into it, while if a lighted candle is inserted in the lower cylinder, it will be extinguished.

Two 2 l. cylinders; siphon; CO_2 supply; candle on wire.

38. Carbon dioxide extinguishes the flame of a candle. — That carbon dioxide is heavier than air and is a non-supporter of combustion is interestingly shown by pouring 2 l. of the gas down into a wooden or metal trough in which five small candles are burning. As the carbon dioxide flows down the trough, the candles are extinguished in rapid succession.

The trough may be made by bending a piece of tin 1 m. long or by tacking two pieces of wood together at right angles. The trough should be inclined at an angle of 45°, and the candles so attached as to burn in an upright position. To prevent an upward current of air, it is best to cut off the lower part of the trough by means of a block of wood or a piece of cardboard (Fig. 126). The candles should be of the smallest size and are placed approximately 15 cm. apart.

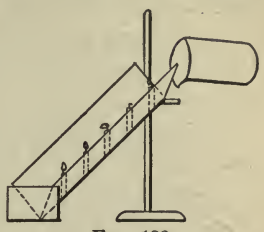

FIG. 126

Trough (Fig. 126); small candles; 2 l. beaker of CO_2.

39. Carbon dioxide in expired air extinguishes a candle flame. — Air from the lungs is collected in a cylinder over water. By taking the last portion of one exhalation, that is, that which has had the longer sojourn in the lungs, a gas quite rich in carbonic acid is obtained, and, indeed, the per cent of this gas will be so great as to cause the flame of a candle, when it is lowered into the cylinder, to be extinguished. By using a flexible rubber tube, which can be pushed clear up the inner wall of the cylinder, the gas may be drawn back into the lungs to become still richer in carbonic acid. In this case the rubber tube should have its end out of water inside the cylinder when the air is withdrawn, to prevent water from being sucked back into the mouth. After removing the cylinder from the pneumatic trough by covering it with a glass plate, the lighted candle is immediately introduced.

300 cc. cylinder; pneumatic trough; candle on wire; long clean rubber tube; glass plate.

40. Preparation of a supersaturated solution of carbon dioxide (soda water). — The technical preparation of a saturated aqueous solution of carbon dioxide may be admirably illustrated on the lecture table by means of a patent device[1] in which small steel capsules containing the liquefied gas are used.

The apparatus consists of a wire or wicker-covered, stout-walled bottle and a special screw-top. As explicit directions are given with each apparatus, it need only be stated here that the bottle is seven-eighths filled with cold distilled water, the top screwed on carefully, the steel capsule inserted in the upper portion of the metallic top, and the gas liberated by screwing down a small screw-cap (Fig. 127). The gas is forcibly driven in a fine jet into the water which, after unscrewing the top, will be found to be supercharged with carbon dioxide. On pouring some of the water into a beaker the excess of gas will ascend in fine bubbles.

FIG. 127

The capsules are especially interesting, and the difference in weight when filled and when empty is very marked. The gas may be withdrawn from the capsule and collected over water by attaching a rubber tube to the end of the jet and very carefully screwing down the small cap, a slow liberation of the gas resulting. The gas issuing from the other end of the rubber tube may be collected in a liter cylinder over water.

The intense cold produced by the evaporation of the liquid may be shown by discharging a capsule in the air,

[1] The apparatus is distributed by the Compressed Gas Capsule Company of New York, and may be obtained in nearly every city in the United States. The small steel capsules are sold under the trade name "Sparklets."

pointing the nozzle down. As the liquid escapes, it forms for a moment a thin fog and the end of the jet becomes extremely cold.

Sparklet apparatus (Fig. 127); liter cylinder; rubber tube; pneumatic trough.

CARBON DISULPHIDE

PROPERTIES

41. Solvent action on fats. — Carbon disulphide dissolves fats readily. A small lump of tallow in a test-tube is covered with carbon disulphide, in which it readily dissolves.

A piece of paper, a portion of which has been greased, is dipped into carbon disulphide and allowed to remain three minutes. On withdrawing the paper and allowing the carbon disulphide to evaporate, the grease spot will have disappeared.

Tallow; greased paper; CS_2.

42. Production of cold by the rapid evaporation of carbon disulphide. — Fifteen cubic centimeters of carbon disulphide are placed in a small flask and covered with 5 cc. of water. A strong blast of air is passed through the mixture, care being taken to provide for the removal of the vapors of carbon disulphide to a flue or hood. In a few moments the water in the flask will be frozen.

If a current of air is passed through carbon disulphide in which a thermometer is placed, the temperature will rapidly fall to 15° below zero.

Small wide-mouthed flask; air-blast; thermometer; CS_2.

43. Comparative inflammability of ether and carbon disulphide. — A glass rod which has been heated at one end is dipped into ether, which is not ignited by it, and then into an evaporating-dish containing carbon disulphide. Suffi-

cient heat will have been retained to ignite the carbon disulphide. The ether and the carbon disulphide are both placed in evaporating-dishes which stand about 30 cm. apart.

Two evaporating-dishes; CS_2; ether.

44. Combustion in oxygen. — One cubic centimeter of carbon disulphide is placed in a deflagrating-spoon and after ignition lowered into a liter cylinder of oxygen. The combustion proceeds with great brilliancy. If the spoon is shaken somewhat so as to swash the liquid upon the hot sides of the spoon, it will evaporate more rapidly, and consequently a larger flame will be obtained.

Deflagrating-spoon; liter cylinder of O; CS_2.

45. Explosion of a mixture of carbon disulphide vapor and oxygen. — A mixture of carbon disulphide vapor and oxygen explodes vigorously, though by selecting a stout-walled cylinder the experiment may be safely performed.

A strong 200 cc. cylinder is filled with oxygen into which 2 cc. of carbon disulphide are poured. The cylinder is covered with a cardboard having a hole in the centre, and is then vigorously shaken. On applying a match to the opening a sharp explosion is obtained. If a heavy-walled cylinder is not at hand, it is advisable to wrap the apparatus with a towel to prevent damage from the explosion.

Stout-walled cylinder; O supply; CS_2.

46. Combustion of potassium. — Potassium burns in the vapor of carbon disulphide, forming polysulphides of potassium.

Carbon disulphide in a 100 cc. flask is heated in a beaker of hot water, and the vapor conducted through a glass elbow into a bulb-tube containing a piece of potassium. Rubber must be avoided in making any of the connections. As soon

as the tube is filled with the vapor of the disulphide, the potassium is heated. The metal burns vigorously.

100 cc. flask; bulb-tube; beaker of hot water; CS_2; K.

47. Combustion of iron. — Fine iron wire, when thrust into the flame of burning carbon disulphide, burns strongly, forming ferrous sulphide.

The vapor of carbon disulphide issuing from the glass elbow in the flask of the preceding experiment is ignited and the size of the flame regulated by the temperature of the water in the beaker. Iron wires which have been twisted together are inserted in the flame. The molten globules of iron sulphide formed may be tested with hydrochloric acid, and the evolution of hydrogen sulphide shown.

100 cc. flask; beaker of hot water; cork and elbow; CS_2; bundle of fine iron wires.

METHANE (MARSH GAS)

48. Preparation from sodium acetate. — Sodium acetate, when heated in the presence of soda-lime, decomposes, liberating methane or marsh gas.

Fifteen grams of fused sodium acetate are pulverized and mixed with an equal weight of fused soda-lime. The mixture is placed in a 100 cc. Jena glass Erlenmeyer flask fitted with a one-holed cork and a wide delivery-tube (Fig. 128). The flask is gradually heated and, after all air has been driven out, the gas is collected in several cylinders for use in the following experiments. The importance of

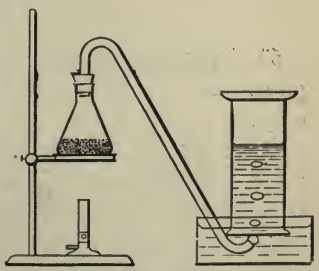

FIG. 128

having water-free materials cannot be too strongly emphasized, as condensed water is likely to break the flask.

100 cc. Jena glass Erlenmeyer flask; pneumatic trough; several cylinders; fused sodium acetate; fused soda-lime.

49. Marsh gas a non-supporter of combustion. — A burning candle thrust into an inverted cylinder of marsh gas is immediately extinguished.

Candle on wire; liter cylinder of CH_4.

50. Explosion with oxygen. — Two volumes of oxygen and 1 volume of marsh gas are introduced into a thick-walled 100 cc. cylinder having a cardboard cover with a 5 mm. hole in the centre. On igniting the mixture, a strong explosion will be obtained. A thick-walled cylinder or a round-bottomed ginger-ale bottle should be used for this experiment, and as a special safeguard the vessel should be wrapped with a towel.

100 cc. stout-walled cylinder filled $\frac{1}{3}$ with CH_4 and $\frac{2}{3}$ with O.

ETHYLENE

51. Preparation from alcohol and sulphuric acid. — Sulphuric acid extracts water from ethyl alcohol, liberating ethylene.

One hundred and twenty cubic centimeters of concentrated sulphuric acid are cautiously poured, with constant stirring, into 30 cc. of ethyl alcohol. After cooling, the mixture is poured into a liter flask one-fourth filled with coarse sand. The flask is fitted with a cork, a safety-tube, and a wide delivery-tube (Fig. 129). On gently heating the mixture a rapid evolution of ethylene is obtained, which may be collected in a large tubulated bell-jar for Ex. 53, and in several

small cylinders for other experiments. As the mixture is liable to char and froth badly, the heat should be very carefully applied. The gas obtained in this reaction is not perfectly pure, and should be washed by conducting it through a solution of sodium hydroxide, if the pure gas is desired. Owing to its solubility in cold water, it is advisable to have the water in the pneumatic trough warm.

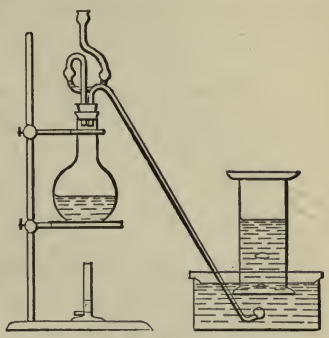

FIG. 129

$$C_2H_6O = C_2H_4 + H_2O.$$

Liter flask; gas washing-bottle; tubulated bell-jar; pneumatic trough with warm water; cylinders; sand; ethyl alcohol.

52. Preparation from ethylene dibromide. — One cubic centimeter of alcohol and 2 cc. of ethylene dibromide are warmed with a few pieces of granulated zinc in a test-tube. The zinc combines with the bromine, setting free ethylene, which may be ignited at the mouth of the tube.

$$C_2H_4Br_2 + Zn = ZnBr_2 + C_2H_4.$$

Alcohol; ethylene dibromide; granulated Zn.

53. Decomposition by heat. — Ethylene is decomposed at a high temperature with the liberation of carbon.

The gas from a gas-holder, or from the bell-jar, Ex. 51, is conducted through a bulb-tube. After all air has been driven out of the apparatus, the bulb-tube is strongly heated in a blast-lamp. By rotating the bulb, a thin mirror-like deposit of carbon may be obtained all over the interior of the bulb.

Bulb-tube; blast-lamp; C_2H_4 supply.

54. Absorption by cold water. — The absorption of ethylene in cold water may be shown by a series of experiments similar to those used to illustrate the solubility of hydrogen sulphide in water (Ex. 17, p. 140).

55. Union with bromine. — Ethylene and bromine unite directly, forming ethylene dibromide.

A 2 l. flask is filled with ethylene, and 2 cc. of bromine are added. The bromine fumes are all absorbed, and, after a thorough shaking, a colorless oil, ethylene dibromide, remains on the bottom of the flask. If an excess of bromine is used, the oil will be colored, and it will be necessary to wash it with a little sodium hydroxide solution to remove the free bromine. The oil, which possesses an ethereal odor, may be poured into a cylinder containing water. The oil is heavier than water.

$$C_2H_4 + Br_2 = C_2H_4Br_2.$$

2 l. flask filled with C_2H_4; cylinder; Br.

56. Absorption by bromine. — The absorption of ethylene by bromine is markedly shown by collecting 100 cc. of the gas in the eudiometer (Fig. 11, p. 26), and allowing one cubic centimeter of bromine to trickle very slowly down the inside of the tube. The bromine in the bulb should be covered with water to prevent the escape of bromine fumes. As the bromine comes in contact with the gas, its color is immediately discharged, the volume of the gas rapidly diminishes, and the oily dibromide formed drops to the bottom of the vessel.

Eudiometer (Fig. 11, p. 26); C_2H_4 supply; Br.

57. Action of chlorine on ethylene. — Chlorine and ethylene unite to form ethylene dichloride, an oily liquid.

Two hundred cubic centimeters of ethylene are introduced

at the pneumatic trough into a 500 cc. cylinder filled with water, and chlorine is then admitted, a few bubbles at a time. It will unite with the ethylene, forming oily drops of the dichloride, which float as a film on the surface of the water, and finally settle to the bottom of the trough. The volume of the gas is diminished.

$$C_2H_4 + Cl_2 = C_2H_4Cl_2.$$

500 cc. cylinder; C_2H_4; Cl supply.

ACETYLENE

58. Preparation from ethylene dibromide and alcoholic potash. — Potassium hydroxide, in alcoholic solution, abstracts hydrobromic acid from ethylene dibromide, with the formation of acetylene.

Alcoholic potash is made by adding a stick of caustic potash to 10 or 15 cc. of alcohol, and pouring off the solution when saturated.

If 3 or 4 cc. of alcoholic potash in a test-tube are heated just to boiling, and a few drops of ethylene dibromide added, a gas is given off, which, on ignition, is readily recognized as acetylene.

$$C_2H_4Br_2 + 2\,KOH = 2\,KBr + 2\,H_2O + C_2H_2.$$

Alcohol; potassium hydroxide (stick); ethylene dibromide.

59. Preparation from calcium carbide. — (*a*) A few pieces of calcium carbide in a dry test-tube are treated with a few drops of water. The evolution of gas is very rapid, and on ignition, the characteristic luminous, sooty flame of acetylene is readily recognized.

$$CaC_2 + H_2O = CaO + C_2H_2.$$
$$CaO + H_2O = Ca(OH)_2.$$

Calcium carbide in small pieces.

(*b*) A more striking decomposition of calcium carbide by water is obtained when a 5 cm. piece is placed on a plate, and a fine jet of water from a wash-bottle or tap is directed on it. The escaping gas can be ignited, and the flame increases as more water is added.

Calcium carbide is now obtainable in the market in tin cans for use with bicycle lamps. It should be preserved in tightly sealed bottles, as it absorbs moisture from the air, and thereby is decomposed.

Wash-bottle ; plate ; CaC_2.

60. Formation by the incomplete combustion of illuminating gas. — In the incomplete combustion of illuminating gas (Ex. 6, p. 345) small quantities of acetylene are formed. This phenomenon is also observed when a Bunsen burner "strikes back."

A Bunsen burner, burning at the base, is placed under the inverted thistle-tube in the apparatus (Fig. 31, p. 62). A sufficient quantity of ammoniacal cuprous chloride solution is placed in the U-tube to cover the bend. A gentle suction is maintained through the apparatus and in a few moments a precipitate of red cuprous acetylide is formed.

Apparatus (Fig. 31, p. 62) ; ammoniacal cuprous chloride solution.

ILLUMINATING GAS (COAL GAS)

61. Preparation. — Coal or wood, when subjected to dry distillation, yields considerable quantities of a gaseous mixture consisting chiefly of hydrogen and methane, together with some of the higher hydrocarbons.

A few grams of pulverized anthracite coal are strongly heated in a hard-glass test-tube. The gas evolved may be ignited at the mouth of the tube, where it burns with a feebly luminous flame.

Bituminous coal, when heated in a test-tube, yields large quantities of a gas which burns at the mouth of the tube with a luminous, smoky flame.

Bits of wood heated in a test-tube yield a gas which burns with a luminous flame.

Hard-glass test-tubes; anthracite and bituminous coal; chips of wood.

62. Preparation by the distillation of bituminous coal. — A 500 cc. Jena glass retort is one-third filled with pulverized bituminous coal, and the neck is thrust into a filter-bottle whose side tube is provided with a glass jet (Fig. 130). The retort is cautiously heated, and the gas evolved is allowed to drive out all air before it is ignited at the jet. The gas burns with a luminous flame. A certain amount of tarry matter is distilled over and condenses with some water in the filter-flask, which should be kept cold by immersion in water.

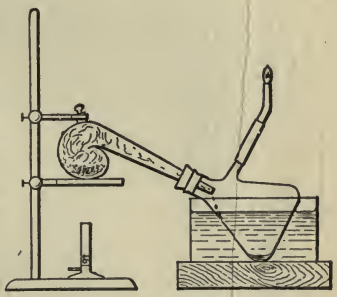

FIG. 130

Apparatus (Fig. 130); 500 cc. retort; sand-bath; filter-bottle; glass jet; pulverized bituminous coal.

63. Soap-bubbles filled with illuminating gas rise in the air. — Soap-bubbles may be blown with illuminating gas, as described in Ex. 13, p. 52. As they ascend, the bubbles may be ignited by touching them with a burning taper on the end of a long stick.

Thistle-tube; candle on long stick; soap solution.

64. Explosion of a mixture of illuminating gas and air. — (*a*) The explosion of a mixture of air and illuminating gas is best shown by means of the following apparatus. The middle neck of a liter three-necked Wolff bottle (Fig. 131) is provided with a cork and a 20 cm. length of combustion tubing, 1 cm. internal diameter. Coal gas is conducted through a glass tube in one of the necks, and the third is closed with a solid cork. All the necks should preferably be as wide as possible. Illuminating gas is conducted through the glass tube, extending to the bottom of the bottle until all air is expelled, and the gas is then ignited as it issues from the large glass tube in the middle neck. On cutting off the gas and simultaneously removing the cork in the third neck, the flame increases in size, though diminishing in intensity, and as soon as enough air has been drawn through the open neck to produce an explosive mixture in the bottle, the flame strikes back through the tube, producing a loud, though harmless, explosion.

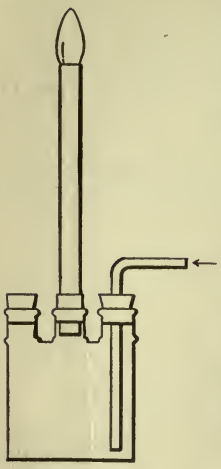

FIG. 131

Apparatus (Fig. 131); 1 liter 3-necked Wolff bottle; combustion tubing.

(*b*) The tubulated bell-jar, with cork and glass tube used in Ex. 29, p. 67, may also be used to produce an explosion of illuminating gas and air.

65. Combustion on platinized asbestos. — Platinized asbestos (Ex. 24, p. 61) is heated to incandescence in a Bunsen

burner. On turning out the gas the asbestos ceases to be luminous. If the gas is now turned on, the asbestos will immediately glow, but the gas is re-ignited with difficulty. If the air-holes at the base of the burner are closed so that no air can enter, the asbestos will again cease to glow, as there is no air in the middle of the gas-jet to cause combustion. On opening the air-holes, the asbestos again becomes luminous, owing to the presence of the mixture of gas and air.

Pt asbestos (Ex. 24, p. 61).

THE NATURE OF FLAME

STRUCTURE OF FLAME

1. The Bunsen burner. — A burner having an air-regulating device for closing the air-holes at the base of the burner-tube should be used in the following experiments. The air-holes should be closed, and then the burner lighted. The large, luminous, flickering flame should be compared with the flame obtained by unscrewing the burner-tube and lighting the gas as it issues from the fine jet (Fig. 132). The ratio of the cross-sections of the tubes through which the gas is issuing in both cases should be noted.

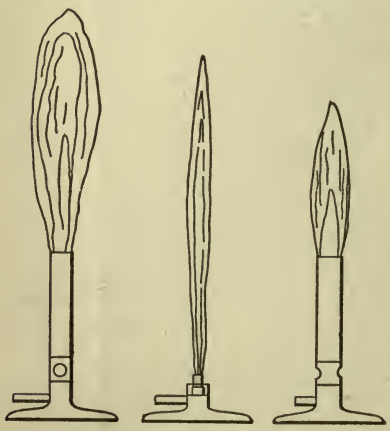

FIG. 132

After replacing the burner-tube, the air-holes still being closed, the burner should again be lighted and the air-holes gradually opened. The variations in the nature of the flame from the flickering, luminous flame to the steady, non-luminous flame should be noticed.

That a certain ratio must exist between the proportion of gas and air mixing in the burner-tube in order to have the flame burn quietly at the top of the burner is observed by gradually turning off the supply of gas, the air-holes being wide open, until the proportion of gas and air in the tube is such as to cause the flame to strike back and burn at the base of the burner. To accentuate the change in position of the flame, the gas should again be turned on full and the character of the flame appearing above the burner-tube noted. By means of a pair of pliers the hot burner-tube may be unscrewed, as before, while the flame is still burning.

2. Striking back of a Bunsen flame. — The striking back of a Bunsen flame may also be shown by means of the apparatus (Fig. 133). (See Ex. 6, p. 345.)

Fig. 133

By turning on a good supply of gas it is easy to get a perfect Bunsen flame burning at the top of the chimney. Several trials will probably be necessary before the best quantity of gas will be found. When the proportions of gas and air entering the base of the chimney are correct, a great blue Bunsen flame will be obtained at the top. By turning down the gas slowly the flame will strike back with a sharp, though harmless report, and burn at the base of the burner.

It is important that the metal part of the burner should not be allowed to become too hot by burning the gas at the base of the chimney, for the hot metal increases the difficulty of securing a well-formed Bunsen flame. If the metal becomes too hot, it is better to wait a few minutes before attempting to relight the burner. As most Argand

burners have a small gas-regulating device at the base, the proportions may well be established before the lecture, and in order to get a good flame it will only be necessary to open the gas-cock on the desk and light the gas.

Argand burner and chimney (Fig. 133).

3. The inner portion of a flame is cool. — (*a*) A striking demonstration of the fact that the inner portion of a flame is cool is made by employing a burner consisting of an ordinary glass funnel 5 cm. across the top, which has a piece of fine copper or brass gauze over the mouth (Fig. 134). The stem of the funnel is connected by means of a rubber tube to the gas-cock. A small heap of gunpowder, about 15 mm. in diameter, is placed in the centre of the wire gauze, care being taken that no small particles of the powder are scattered on the gauze away from the middle of the heap. After protecting the apparatus from strong draughts, the gas is turned on and a match brought down from above until the gas is ignited. It burns with a large flame, and the gunpowder remains on the wire gauze unconsumed. Ordinary matches may be thrust suddenly through the flame, and their heads laid on the heap of gunpowder, without being ignited. On slowly turning down the gas, the flame will diminish in size and soon play over the surface of the gunpowder and cause its ignition.

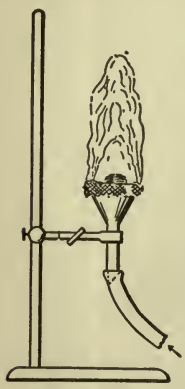

Fig. 134

In repeating the experiment, care should be taken to allow the gauze covering to become perfectly cold, and *in no case must the powder be added from the bottle or other container*, but rather from a small piece of paper. In introducing the

phosphorus matches, care should be taken not to disturb the heap of gunpowder and scatter the grains. While the explosion of such a small quantity of gunpowder is not dangerous, the eyes and face should be protected from any possible accident.

5 cm. funnel; fine wire gauze; gunpowder; shields or eye-glasses.

(*b*) A pin or needle is thrust through a match just below the head. On allowing the match, supported on the needle, to hang down the tube of a Bunsen burner, the gas may be lighted and the match remain unlighted in the centre of the non-luminous flame. The match, to avoid touching the edge of the cone, and thereby becoming ignited, should be so placed that it is as near as possible to the centre of the inner cone. A Bunsen burner giving a perfect flame is necessary for this experiment.

4. Pictures of flames with asbestos paper. — An interesting study of the temperature zones of an ordinary Bunsen flame may be made by depressing for a few moments a piece of paper on the flame. The hotter portions of the flame will soon char the paper, while the paper in the cooler zones will not become darkened, the intensity of the charring showing the intensity of the heat. It will thus be seen that the centre of the flame is comparatively cool, as a small circle 7 mm. in diameter will be unburnt.

The most satisfactory paper for this experiment is ordinary thin asbestos paper. In the manufacture of this paper certain quantities of grease or oil are incorporated with the asbestos. On thrusting the paper into the flame the heat chars the oil, producing a blackening of the paper, and one can readily determine, by noticing the intensity of the color, the relative temperatures of different parts of the flame. Asbestos paper possesses another advantage, *i.e.*, it is not com-

bustible. If a 15 cm. square piece of thin asbestos paper is held vertically in the flame, a remarkably good picture of the cross-section of the flame may be obtained. Some slight degree of skill is necessary to avoid heating the paper too hot, and thereby charring too much of it, though the eye readily perceives when the paper should be withdrawn from the flame. Pictures of the flame in almost any position may be made by using this paper (Fig. 135).

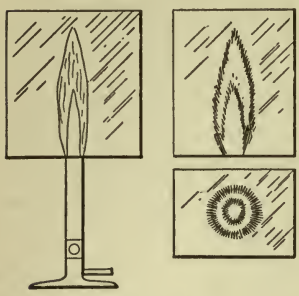

FIG. 135

5. Pictures of a flame on metallic copper. — The beautiful colors formed in the oxidation of copper may be advantageously used to form a picture of a Bunsen flame.

A 20 cm. square of thinnest sheet copper is carefully cleaned and polished. The sheet is then held in the Bunsen flame, as described in the preceding experiment. Immediately the copper will take on in colors the outline of the flame, but on account of the metal's great conductivity of heat, the sheet must be heated for only a few moments.

In case the experiment is to be repeated, the picture can be instantly effaced by washing with a few drops of potassium dichromate solution to which some sulphuric acid has been added.

Pictures of the flame in numerous positions can be readily obtained by this method.

Thin sheet copper; $K_2Cr_2O_7$ solution.

6. Ignition of the gas arising from a recently extinguished candle. — That the candle flame is due chiefly to the burn-

ing of the gas formed by the action of heat on the solid portion is shown by blowing out the candle and holding the match 1 or 2 cm. above the wick, when it will be seen that the gas or smoke rising from the wick will catch fire and run back, communicating its flame to the wick. To show the ignition of the gas more strikingly, the candle is placed inside of an ordinary Argand lamp chimney (Fig. 136). The chimney must be raised from the table to allow a good current of air to enter, and sufficient air must be furnished to allow the candle to burn steadily. On blowing out the candle the gas will rise, and a match held at the top of the chimney will set fire to the gas and ultimately to the candle as described above. It is not unusual to have the flame descend some 6 or 8 cm. to the wick. Instead of a match a flame from a blowpipe jet or a glass tip may be directed across the top of the chimney, and the distance from the top of the chimney to the top of the wick measured as the distance that the flame has travelled.

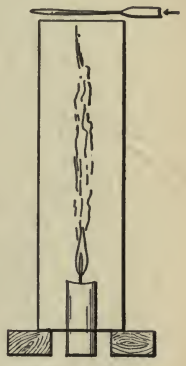

FIG. 136

Argand lamp chimney; candle.

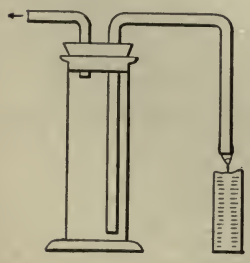

FIG. 137

7. Collection of combustible gases from the interior of a candle flame. — A 300 cc. glass cylinder is fitted with a two-holed rubber stopper carrying a short glass elbow and a long glass tube reaching to the bottom of the cylinder and bent over on the outside so as to descend to the wick of a wax candle (Fig. 137). The

candle is so held that the tip of the wick just touches the end of the long glass tube. On lighting the candle and applying a gentle suction to the glass elbow, the unburned gases generated by the heat of the candle are drawn over into the cylinder. After a few moments, sufficient gas will have been collected to burn when the cork is removed and a flame applied. If the cylinder is not completely filled with the gas, enough air may remain to cause a slight explosion.

300 cc. cylinder; suction-pump; candle.

8. Cooling a Bunsen flame with wire gauze. — When a Bunsen flame is allowed to play upon a piece of wire gauze, the heat is conducted from the flame along the wire so rapidly as to prevent the gas that rises through the meshes from being ignited.

A piece of wire gauze is suddenly depressed on the top of the inner cone of a Bunsen flame. The flame will remain entirely on the lower side of the gauze, though in a few moments, as the wire becomes heated, an ignition of the gas above the gauze results.

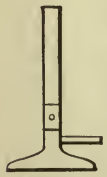

Fig. 138

The experiment may be varied by turning on the gas without lighting it, holding a piece of wire gauze horizontally about 4 cm. above the top of the burner, and applying a flame from above. The gas will burn on top of the gauze, and it will be impossible to generate sufficient heat to ignite the gas below (Fig. 138).

This cooling effect of wire gauze finds practical use in the Davy safety-lamp.

9. The Davy safety-lamp. — The use of the Davy safety-lamp in the presence of explosive gaseous mixtures is well illustrated by lowering it into a mixture of ether vapor and air.

A 2 l. beaker is used to hold the explosive mixture obtained by pouring 5 cc. of ether vapor into the beaker and then covering its mouth with a cardboard cover. A Davy lamp is lighted (care being taken that the flame is not too high) and carefully lowered into the beaker. As it comes in contact with the gaseous mixture, a flame may be seen playing for a few seconds inside the wire gauze surrounding the lamp. As the lamp is lowered, the flame is extinguished, owing to the deficiency in oxygen, and the gaseous mixture is not ignited.

2 l. beaker; cardboard cover; Davy safety-lamp; ether.

10. Increased illumination of a Bunsen flame by the introduction of particles of carbon. — (*a*) The incandescence of small particles of carbon which are caused to pass through a Bunsen flame renders it luminous.

The simplest method of introducing the particles of carbon is to rub together two pieces of charcoal near the air-holes at the base of the burner. The fine charcoal dust will be drawn up into the tube and cause the flame to become luminous.

Two pieces of charcoal.

(*b*) By introducing very finely divided carbon into a colorless Bunsen flame a luminous flame is obtained. An extremely finely divided form of carbon is obtained as lampblack from the flame of burning turpentine, and if a current of air ladened with the soot from a flame of this nature is conducted into a Bunsen flame, the flame is rendered luminous.

Turpentine is burned in a small lamp made by thrusting a short piece of glass tubing into a cork, having a slit cut in one side, fitting the mouth of a small bottle. A piece of round cotton wick is drawn through the glass tube, and the

bottle is one-half filled with turpentine. On lighting the free end of the wick, a small smoky flame will be obtained.

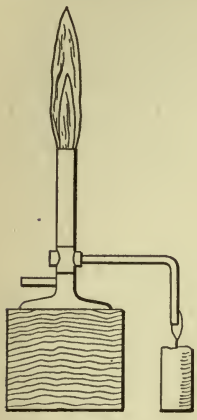

Fig. 139

A 12 cm. length of combustion-tubing is so clamped that it serves as a chimney to deflect the products of combustion into the lower part of a Bunsen flame. The column of soot rising through the combustion-tube enters the Bunsen flame and there becomes heated to incandescence.

The products of the incomplete combustion of a candle may be conducted into the base of a Bunsen burner by means of a glass elbow thrust into one of the air-holes (Fig. 139). On lighting the Bunsen burner, sufficient draft will be obtained to conduct the products of combustion of a burning candle, the tip of whose wick is thrust into the lower end of the glass elbow, up into the Bunsen flame. The flame is rendered luminous.

Turpentine lamp; 12 cm. length of combustion-tubing; glass elbow; candle.

11. Carburetting a flame of hydrogen. — The introduction of solid particles of carbon into a non-luminous flame may be effected by introducing vapors of carbon compounds which are decomposed readily by heat, liberating finely divided carbon.

A current of hydrogen is conducted through a test-tube containing some cotton-batting and fitted with a three-holed cork (Fig 140). In one hole is a glass elbow, extending to the bottom of the test-tube, through which the hydrogen enters. The hydrogen issues through a bent glass tube serving as a jet in another hole. A small dropping-funnel

containing benzine is fitted in the third hole. After all air is driven out of the apparatus, the hydrogen is ignited at the jet, where it burns with a non-luminous flame. A platinum tip should be used. On opening the stop-cock of the dropping-funnel and allowing a few drops of benzine to fall upon the cotton, the flame immediately becomes luminous. The benzine vapor carried along with the hydrogen is decomposed by the hot hydrogen flame, setting free carbon, a part of which becomes heated to incandescence. The actual liberation of carbon is proved by the soot deposited on a cold porcelain dish depressed upon the luminous flame. A dish depressed upon the hydrogen flame produces no deposit.

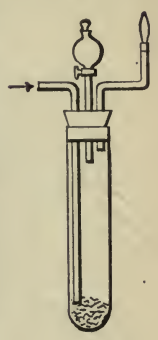

FIG. 140

A similar apparatus may be used to illustrate the carburetting of ordinary coal gas, in which case, however, it is better to replace the platinum tip by a regular gas-burner. Coal gas issuing from a jet saturated with benzine vapor burns with a much more luminous flame than the uncarburetted gas, as may be readily seen by holding an ordinary gas-flame beside it.

Large test-tube ; three-holed cork ; dropping-funnel ; cotton-batting ; Pt jet ; H supply ; benzine.

12. Luminosity of a Bunsen flame increased by finely divided nickel. — In Ex. 9, p. 418, carbon monoxide, saturated with the vapor of nickel tetracarbonyl, is conducted into the air-holes of a Bunsen burner. The vapor mixes with the illuminating gas, and the heat of the flame is sufficient to decompose the tetracarbonyl, setting free finely divided nickel which is burned in the flame, imparting to it an intense luminosity.

Waste gases from Ex. 9, p. 416.

13. Increase in brilliancy of a non-luminous flame by heating. — A piece of platinum foil, at least 15 cm. long, is rolled in the form of a tube, bound with some pieces of platinum wire, and slipped over the end of a Bunsen burner, forming an extension to its main tube. The burner is clamped in an inclined position, and on opening the air-holes at the base, the flame is so regulated as to appear just non-luminous at the top (Fig. 141). On heating the platinum tube with a strong burner, the issuing gas at the end of the platinum tube will be seen to have acquired a decided luminosity. It is advisable to heat the platinum thoroughly before using it for this experiment, in order to burn off all dust, which would otherwise color the flame.

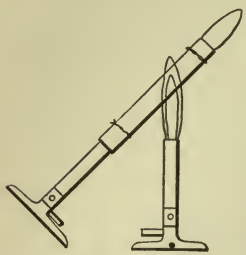

FIG. 141

Platinum foil 15 cm. long.

14. Luminosity of a flame decreased by dilution with an indifferent gas. — Illuminating gas, when diluted with carbon dioxide, loses its illuminating power.

Carbon dioxide from a Kipp generator is introduced into one of the air-holes in the base of a Bunsen burner by means of a small cork and a glass tube (Fig. 139). The other air-hole is closed with a cork. Illuminating gas is conducted through the burner and ignited at the top, where it burns with a flickering, luminous flame. Carbon dioxide is then slowly admitted, with the result that the flame diminishes in luminosity until finally it becomes blue.

The same effect may be obtained by using the chromium oxychloride-hydrogen, in place of the illuminating-gas, flame.

By heating the diluted gas with the platinum extension-tube of Ex. 13, the luminosity may again be restored.

Bunsen burner with cork and tubes in air-hole; CO_2 generator; Pt tube (Ex. 13).

15. Effect of the dilution of air on a luminous flame. — The diminution in luminosity in a flame, resulting from the dilution of the combustible gas by an indifferent gas, may also be obtained by diluting the atmosphere with a non-combustible gas such as carbon dioxide. An artificial atmosphere, containing one-third carbon dioxide, is prepared by filling two-thirds of a large bottle with air and the remaining volume with carbon dioxide. Illuminating gas is conducted through the recurved jet (Fig. 41, p. 85) and ignited. A luminous flame, 3 or 4 cm. high, is obtained, and the jet is lowered into the artificial atmosphere of air and carbon dioxide. The luminosity of the flame disappears, though by withdrawing the jet it can be seen that the gas has not been extinguished.

Hydrogen, burning from the recurved jet having a platinum tip on which a small amount of sodium chloride solution has been placed, burns in the air with a brilliant yellow flame. When lowered into the diluted atmosphere, the luminosity disappears.

A candle lowered into such an atmosphere is immediately extinguished.

Large bottle containing one-third CO_2, two-thirds air; jet (Fig. 41, p. 85); NaCl solution; H generator; candle on wire.

RECIPROCAL COMBUSTION

1. Combustion of oxygen in hydrogen. — (*a*) A jet of oxygen will burn as well in an atmosphere of hydrogen as a jet of the latter gas will burn in the presence of air or

oxygen. That the terms "combustible" and "supporter of combustion" are only relative is well shown by an experiment in which oxygen, ordinarily considered non-combustible, burns in an atmosphere of hydrogen which extinguishes the flame of a candle.

A liter cylinder filled with hydrogen is held mouth downwards and the gas ignited at its mouth. A slow stream of oxygen is passed through a glass tube which is carefully thrust up into the jar of hydrogen (Fig. 42, p. 85). As the tip of the glass tube passes the burning gas the oxygen is ignited. If a small platinum tip is provided, the flame will be seen to be colorless. If the oxygen tube is withdrawn, the flame will be extinguished as soon as it leaves the atmosphere of hydrogen. The tube is again introduced, and the gas will continue to burn until the hydrogen is consumed. To prevent the possibility of the formation of an explosive gaseous mixture, care must be taken that the oxygen flame is not extinguished by accidental pressure on the rubber tube conducting the oxygen. Should the flame be extinguished, the glass tube must be immediately withdrawn and the jar refilled with hydrogen, after expelling all the remaining gas.

Liter cylinder of hydrogen ; current of oxygen.

(*b*) The liter cylinder of hydrogen may be advantageously replaced by an apparatus for furnishing a constant supply of hydrogen. The simplest form of apparatus consists of a small lamp chimney vertically clamped with a one-holed cork fitted into the upper end. A slow stream of hydrogen is passed through a glass tube in this cork, and the gas lighted at the lower end of the chimney. The glass tube conducting the oxygen is now thrust into the lamp chimney from below, with all the precautions described above.

Kipp H generator ; lamp chimney ; current of oxygen.

2. Continuous combustion of oxygen in hydrogen. — Oxygen may be made to burn continuously in an atmosphere of hydrogen by means of the apparatus, Fig. 142.

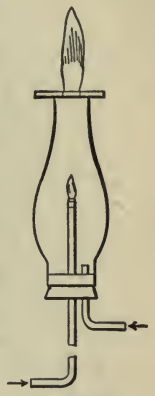

FIG. 142

Hydrogen is conducted through the elbow in the cork at the bottom of the lamp chimney, and is ignited at the opening in the cover at the top. A long glass elbow, which may be easily raised and lowered, is thrust through the second hole in the cork until the end is just on a level with the cover. After hydrogen has displaced all air in the apparatus, and has been lighted as it issues from the top, a gentle current of oxygen is passed through the long glass elbow, and is seen to burn in the interior of the hydrogen flame. The tube may be then slowly lowered, and it will be seen that the oxygen burns in the atmosphere of hydrogen. As the heat of this flame is very intense and is liable to melt the glass, it is advisable to provide for the glass tube a platinum tip, such as is described in Ex. 5, p. 183.

The greatest precaution must be exercised to prevent the formation of a gaseous explosive mixture in the interior of the lamp chimney; and in case the inner flame is extinguished by accidental pressing or kinking of the rubber tube, the oxygen supply must be immediately cut off, as otherwise a dangerous explosion might easily occur.

Apparatus (Fig. 142); lamp chimney; cork and tubes; H and O supply.

3. Combustion of air in illuminating gas. — A simple apparatus for showing this phenomenon consists of an Argand lamp chimney (Fig. 143) fitted with a large cork, car-

rying a small elbow and a 10 cm. length of combustion-tubing. The combustion-tubing should be provided with a platinum tip, made by rolling a piece of platinum foil in such a manner that it will snugly fit the interior of the tube. The chimney is then clamped in an upright position with the open end uppermost, and a cover of thick asbestos card, or better, a brass cap, is fitted on the top of the chimney. A 2 cm. opening should be made in the centre of the cap.

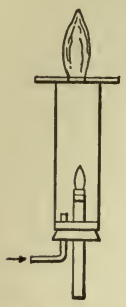

Fig. 143

On closing the opening in the cap and admitting illuminating gas through the elbow, the air in the lamp chimney is soon driven out, and the gas may be ignited as it issues from the lower end of the combustion-tube. On opening the orifice in the cap, the gas rises and the flame recedes through the combustion-tube, and appears at the platinum jet inside the chimney. The escaping gas may be lighted at the top of the chimney and there simultaneously appears a flame of gas burning in air and a flame of air burning in gas. It may be necessary to choke the piece of combustion-tubing by means of a small cork with a slit cut in one side, to prevent too large a volume of air from entering the chimney through the tube. By properly regulating the supply of coal gas and the admission of air, a flame 2 or 3 cm. high is easily obtained.

While the two flames do not appear markedly different, it will be found on thrusting a piece of paper or a visiting card on the end of a wire through the opening at the top of the chimney into the inner flame, that only that portion of the card will be burned which is actually in the flame itself. By carefully inserting the card, a picture of the flame may be obtained by charring the card.

Apparatus (Fig. 143); lamp chimney ; corks and tubes ; asbestos or brass cap.

4. Combustion of air in hydrogen. — By using an apparatus similar to that shown in Fig. 143, in which, however, the large glass tube in the cork has a U rather than a straight form, the combustion of air in hydrogen may be well studied. The apparatus is shown in Fig. 144, and consists of a lamp chimney provided with a two-holed cork at the bottom, carrying a small glass elbow and a large U-tube 1 cm. in diameter. Each end of the U-tube should be provided with a platinum tip (Ex. 3). A cork should be inserted in the top of the chimney and hydrogen admitted. After all the air is expelled, the hydrogen escaping from the outer limb of the U-tube is ignited. On removing the cork from the top of the chimney, the flame recedes through the U-tube, and soon appears burning at the other end inside the chimney as a flame of air burning in an atmosphere of hydrogen. On again inserting the cork in the top of the chimney, the hydrogen will escape through the U-tube, and the flame recede and appear as a jet of burning hydrogen at the outer end of the U-tube. The operation may be repeated as often as is desired. The best results are obtained when the flame at the open end of the U-tube is not more than 1 or 2 cm. high.

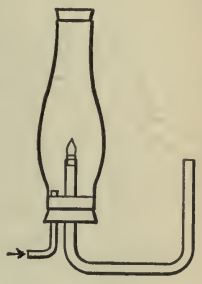

FIG. 144

The operation is even more satisfactory when illuminating gas is used in place of hydrogen, since it has the advantage of giving a luminous flame.

Apparatus (Fig. 144); lamp chimney; large U-tube; corks; H supply.

5. Reciprocal combustion of illuminating gas and air. — An interesting modification of the experiment illustrating the relative combustibility of gas and air is made by means

of the apparatus (Fig. 145). An ordinary lamp chimney is fitted with a two-holed cork carrying a glass elbow and a glass tube, 7 mm. internal diameter. The cork is clamped in a vertical position and illuminating gas is conducted through the small glass elbow and ignited. The chimney is then placed over the cork. The flame will continue to burn, a liberal supply of air entering the lamp chimney through the large glass tube. On turning on more gas, however, the flame will soon ascend and burn at the asbestos cover on the upper part of the chimney, while a current of air drawn through the large glass tube will burn at the end of the tube inside the chimney. A small glass tube is drawn out to a long, fine jet, through which illuminating gas is conducted and allowed to burn.

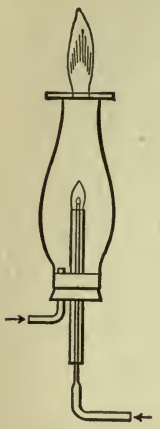

Fig. 145

The small flame of burning illuminating gas may then be thrust through the large tube into the centre of the flame of air burning in the chimney. The illuminating gas will still continue to burn, and there is simultaneously obtained a flame of illuminating gas burning at the top of the chimney in the air, a flame of air burning in illuminating gas inside the chimney, and a flame of illuminating gas burning inside the flame of air. That this last flame will burn only in air is seen by thrusting the glass tube through the air flame into the atmosphere of illuminating gas. On withdrawing the tube the gas is again ignited and continues to burn in the air. It is important that the long, fine tube be of small diameter, as a large tube will so obstruct the passage of air as to make it impossible to secure a good flame. If the illuminating gas admitted through the glass elbow is now slowly turned

off, the flame at the top of the chimney will diminish in size and finally recede into the chimney and burn at the end of the glass elbow as at first.

Apparatus (Fig. 145); lamp chimney; asbestos disk with 2 cm. hole; cork and tubes; long glass jet.

6. Combustion of air in illuminating gas. — By means of the apparatus (Fig. 146) the combustion of air in illuminating gas may be very easily shown.

An Argand gas-burner is provided with a large chimney, such as is shown in the figure. This form of chimney is not designed for use on an Argand gas-burner, but the constriction just above the flame makes it especially desirable in connection with this experiment. A piece of sheet copper or galvanized iron, with a 7 mm. hole in the centre, is laid over the top of the chimney after the gas is lighted and burning with a flame approximately 2 cm. high. When the disk is placed over the chimney, the gas smokes inside the chimney, the flame recedes, and may be seen to be burning at the central air-draft instead of at the ring where the gas issues through the burner.

Fig. 146

By regulating the supply of gas, a flame some 1 or 2 cm. high may be obtained. The gas issuing from the hole in the disk may be lighted, and it will burn with a feebly luminous flame.

It is thus possible to have a flame of air burning in illuminating gas at the base of the burner and a flame of gas burning in air at the top. This is one of the simplest methods of showing the phenomenon of reciprocal combustion.

Argand burner and chimney (Fig. 146); copper or galvanized iron disk with 7 mm. hole in centre.

7. Combustion of oxidizing agents in an atmosphere of hydrogen. — A number of substances furnishing a supply of oxygen may be made to burn in an atmosphere of hydrogen.

The hydrogen is held in a liter bell-jar clamped mouth downwards, or use may be made of a lamp chimney through which a stream of hydrogen is sent from the top, issuing at the bottom. Potassium chlorate is placed in a small porcelain crucible suspended in the loop of a stout iron wire long enough to extend up into the jar or lamp chimney. The potassium chlorate is then melted and strongly heated. When oxygen begins to be liberated it is suddenly thrust through the burning hydrogen at the mouth of the bell-jar or lamp chimney. The salt will burn with intense brilliancy with the characteristic color of potassium salts. By the use of strontium or barium chlorates the flame will be colored respectively red and green. An intense yellow flame may be obtained by using a mixture of four parts potassium chlorate and one part sodium nitrate.

Nitric and iodic acids, both rich in oxygen, burn in an atmosphere of hydrogen when heated. A small quantity of fuming nitric acid is placed in a crucible and heated to boiling. Without waiting long enough to allow much of the oxides of nitrogen to escape, the crucible is thrust into the atmosphere of hydrogen, where it will burn. A small quantity of iodic acid placed in a crucible and heated very hot will burn on being introduced into the hydrogen atmosphere, large quantities of iodine being liberated and deposited on the walls of the jar.

FIG. 147

As the intense heat of the hydrogen burning at the mouth of the jar or lamp chimney is likely to break the glass on long-continued heating, it is advisable to pass the hydrogen through the bottom of an ordinary Argand

lamp chimney clamped in a vertical position, fitted with a one-holed cork and a glass elbow at the bottom, and covered at the top with a disk of thick asbestos with a 2 cm. opening in the centre (Fig. 147). In this form of apparatus the hydrogen burns at the opening of the asbestos, causing no undue heating of the glass. The materials to be introduced into the hydrogen atmosphere must, however, be held in a crucible suspended from a long iron wire capable of being lowered through this opening.

Lamp chimney; H generator; deflagrating-spoon; apparatus (Fig. 147); asbestos disk; $KClO_3$; $Ba(ClO_3)_2$; $Sr(ClO_3)_2$; $NaNO_3$; fuming HNO_3; I_2O_5.

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METALS

The division of the elements into the two groups, non-metals and metals, though more or less arbitrary, is here made in the customary manner.

The size of this book will not permit of an exhaustive list of experiments on the metals, and hence those here selected are to be looked upon only as suggestive additions to the usual precipitations.

SODIUM

1. Metallic lustre. — Metallic sodium, as ordinarily obtained in the market, is covered with a crust, and the metal must be cut in order to show the metallic lustre.

A piece of the metal is laid on dry filter-paper to remove the excess of naphtha and then cut with a knife. The freshly cut surface shows the bright metallic lustre of this element, but almost immediately, *i.e.*, in a very few seconds, becomes covered with a tarnish.

A piece of sodium from which the thick outer crust has been removed may be freed from all tarnish by immersing it for a few moments in absolute alcohol. The sodium reacts with the alcohol somewhat, and the outer surface is entirely freed from oxides. The piece of sodium must then be thrust under naphtha, where its lustre will be preserved for some time.

Absolute alcohol ; naphtha ; Na.

2. Melting the metal. — Sodium may be readily melted if several small pieces are heated under kerosene in a large, dry test-tube. The metal melts at 94° C. to a silver-colored liquid, which preserves its lustre for some time if well covered with kerosene.

If the test-tube is allowed to become cold, the metal solidifies, taking the form of the interior of the tube.

Large, dry test-tube ; kerosene ; Na.

3. Action of sodium on water. — The action of sodium on water with the liberation of hydrogen and the formation of sodium hydroxide is best shown by means of the apparatus (Fig. 148). A glass tube, 20 cm. long and 9 to 10 mm. internal diameter, is clamped in a vertical position with its lower end dipping 3 cm. below the surface of water in a 300 cc. cylinder. The water should about three-fourths fill the cylinder and should contain a few drops of phenol-phthalein solution. A piece of sodium is pressed into a spherical mass not more than 4 mm. in diameter. In preparing this pellet of sodium it is important that the hands should not be wet or too moist from perspiration. On dropping the sodium into the vertically clamped tube the decomposition of the water is immediately effected, and in a few moments hydrogen may be ignited at the upper end of the tube. The sodium hydroxide obtained in the reaction, being heavier than water, settles to the bottom of the cylinder in currents and, by reason of its alkalinity, imparts a strong color to the phenol-phthalein solution.

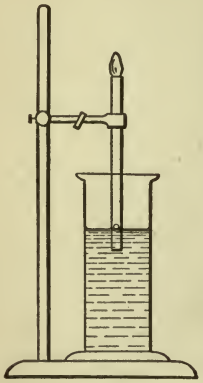

FIG. 148

If care is taken to prepare a spherule of sodium, it will remain in the centre of the cup formed on the surface of the water by the capillarity of the tube and regularly evolve hydrogen. At times, however, especially if the sodium is not spherical in form or if the interior of the glass tube is wet above the level of water in the cylinder, the sodium will stick to the glass, and a slight explosion will occur.

Apparatus (Fig. 148); 20 cm. length glass tube 10 mm. diam.; phenol-phthalein solution; Na.

4. Color change of sodium peroxide by heat. — One gram of sodium peroxide is heated in a test-tube. The powder undergoes a marked change in color from white to yellow. This change in color, however, indicates no chemical change, as the introduction of a glowing splinter fails to detect any evolution of oxygen, and, on cooling, the yellow disappears.

Sodium peroxide; splinter.

5. Oxidizing action of sodium peroxide. — (*a*) *With phosphorus.* — A very small quantity of sodium peroxide is carefully mixed on paper with an equal volume of red phosphorus. The mixture is then wrapped in a powder paper (Ex. 25, p. 248), placed on an anvil, and struck with a hammer. A sharp report is heard. (Gauntlets.)

Hammer and anvil; powder paper (Ex. 25, p. 248); gauntlets; Na_2O_2; red P.

(*b*) *With carbon.* — A small quantity of powdered charcoal is mixed with an equal volume of powdered sodium peroxide and gently heated. The oxidation is very vigorous.

The sodium peroxide should first be placed in the test-tube and then the charcoal added and the mixture well shaken. At times the reaction will take place even in the cold.

The mixture of charcoal and sodium peroxide may be caused to explode by adding one drop of water. The experiment should be performed behind a glass screen.

A few drops of nitrobenzene are allowed to fall upon 1 or 2 g. of powdered sodium peroxide in a dry test-tube. On gently heating the tube an explosion is obtained.

Glass screens; Na_2O_2; nitrobenzene; powdered charcoal.

6. Supersaturation of sodium sulphate solution. — The phenomenon attending the disturbance of a supersaturated solution is well shown by the solution of sodium sulphate.

Two hundred grams of crystallized sodium sulphate are heated in a 500 cc. flask until the salt has completely melted. A plug of cotton should then be placed in the mouth, and the flask allowed to stand until cold. If the operation is performed with care, the salt will remain in the liquid condition. On removing the cotton plug, and dropping a small crystal of sodium sulphate into the melted liquid, the contents of the flask immediately solidify.

If the bulb of the ether thermometer (Ex. 73, p. 174) is inserted in the liquid, the rise in temperature resulting from the solidification of the salt will cause the ether to boil and escape from the mouth of the tube.

The solidification of the supersaturated solution may also be accomplished by rapidly pouring it into a crystallizing dish.

By agitating it with a glass rod, the liquid may be caused to solidify while in the flask.

500 cc. flask; cotton-batting; ether thermometer (Ex. 73, p. 174); crystallized Na_2SO_4.

7. Freezing mixture of sodium sulphate and hydrochloric acid. — Crystallized sodium sulphate, when mixed with hydrochloric acid, produces a lowering in temperature amounting to nearly 30° C.

Eighty grams of the crystallized salt are finely pulverized and mixed in a flask with 40 cc. of concentrated hydrochloric acid. If the flask is placed upon a few drops of water on a block, the water will be frozen and the flask cemented to the wood. The acid must be previously cooled to 10° C.

Flask; block of wood; $Na_2SO_4 + 10H_2O$; con. HCl.

8. Formation of metallic silicates in a solution of sodium silicate. — The metallic silicates may be formed in a solution

of sodium silicate by dropping crystals of the various salts into the liquid. As the silicate forms it spreads out in a treelike structure.

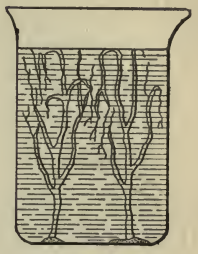

FIG. 149

A liter beaker is filled with a solution of sodium silicate having a specific gravity of 1.10. Crystals of cobalt nitrate, nickel sulphate, uranium nitrate, manganese sulphate, ferrous sulphate, and copper sulphate are allowed to fall in the beaker and rest on different parts of the bottom (Fig. 149). The formation of the silicates requires some time, though frequently marked indications of their formation may be observed at the end of half an hour. The beaker should be placed in a quiet place, and not disturbed until the next exercise.

Large beaker; water-glass solution (sp. gr. 1.10); crystals of $Co(NO_3)_2$, $NiSO_4$, $UrO_2(NO_3)_2$, $MnSO_4$, $FeSO_4$, $CuSO_4$.

POTASSIUM

1. Vaporization. — Potassium, when strongly heated in a glass test-tube, yields a green vapor.

A small piece of potassium, carefully cleaned and dried, is placed in a dry test-tube and strongly heated. The metal is vaporized and rapidly attacks the glass, liberating the silicon. The interior of the tube becomes blackened.

The color of the potassium vapor may be better seen if a piece of clean, dry potassium is heated in a bulb-tube through which a current of dry hydrogen is passed. After making sure that the bulb is filled with hydrogen and that all air is expelled, the potassium is strongly heated in a blast-lamp, the supply of hydrogen being cut down to a minimum. If the heat is supplied quickly, the globule of molten potassium bursts, filling the whole bulb with a green vapor.

The current of hydrogen may be increased, and the issuing gas will burn with the violet flame of potassium.

H generator ; dry test-tube ; dry bulb-tube ; blast-lamp ; K.

2. Union of potassium and bromine. — Potassium and bromine unite with explosive violence to form potassium bromide.

A 3 mm. piece of potassium is carefully dried between filter-paper and dropped into a test-tube containing a few drops of bromine. As the reaction is so violent, special precautions are necessary when performing this experiment.

The test-tube must be placed inside a wide-mouthed bottle (Fig. 109, p. 262) to collect the bromine in case of accident. The closed end of the test-tube is thrust through a hole in a cork or piece of cardboard. A large inverted funnel, with a cork in the stem, is suspended by a ring-stand 15 cm. directly above the tube, to prevent pieces of potassium or drops of bromine from coming out of the tube. Finally, the hands should always be protected with gauntlets.

$$2\ K + Br_2 = 2\ KBr.$$

Apparatus (Fig. 109, p. 262); wide-mouthed bottle; cardboard; funnel; gauntlets; K; Br.

3. Union of potassium and iodine. — When heated, potassium and iodine unite directly with explosive violence.

A 3 mm. piece of potassium is gently warmed in a clean, dry test-tube, with a crystal of iodine. The elements unite with an explosion.

Dry test-tube; K; I.

4. Use of potassium nitrate in touch-paper. — Unsized paper drenched in concentrated potassium nitrate solution and then dried finds much use as touch-paper for igniting combustible mixtures. A quantity of the paper should be prepared for general lecture-table use.

A solution of potassium nitrate, saturated at the temperature of the room, is placed in a small beaker. Strips of filter-paper are then thoroughly moistened with the solution, hung up, and allowed to dry (not by a flame). If the solution is saturated at the boiling temperature, an excess of nitre will be crystallized on the surface of the paper in such a way as to be continually rubbing off.

On igniting one end of a strip of the dried paper the combustion will slowly proceed till the paper is completely

consumed. The fire cannot be extinguished by blowing on it.

By means of a fine camel's-hair brush a letter or word is written on a sheet of unsized paper with a concentrated solution of potassium nitrate. After the paper has become thoroughly dry and suitably suspended, a match touched to one corner of the word will cause a lively combustion, which will follow the line of the word and ultimately burn it out. A brush marking lines not over 3 mm. in width should be used, as otherwise there is danger of the whole paper taking fire.

Unsized paper; fine camel's-hair brush; saturated solution of KNO_3.

5. Preparation of gunpowder. — Thirty grams of finely powdered potassium nitrate, 5 g. of powdered charcoal, and 5 g. of sulphur flowers are intimately mixed on paper. Three-fourths of this mixture is placed on a plate or a piece of asbestos paper and ignited in the hood. When the mixture is ignited, it burns brightly, leaving a residue which contains potassium sulphide. On moistening the residue with hydrochloric acid, hydrogen sulphide is liberated.

The value of pressing and granulating gunpowder is markedly shown by the following experiment: The remainder of the above mixture is placed on one square of asbestos and an equal quantity of gunpowder is placed on a second square. Two strips of touch-paper are cut of equal length and inserted in the two heaps of powder. After placing the asbestos squares in the hood the touch-paper is ignited. The commercial gunpowder burns with an instantaneous flash, while the mixture burns much more slowly.

Asbestos paper; touch-paper; gunpowder; KNO_3; powdered charcoal; S flowers.

6. Combustion of phosphorus in potassium chlorate. — A depression is made in the top of a small heap of finely pulverized potassium chlorate on asbestos paper. A 3 mm. piece of well-dried phosphorus is laid in the depression and, after placing the asbestos plate in a strong draft, the phosphorus is ignited by means of a candle or taper on the end of a long stick. The phosphorus burns fiercely in the oxygen liberated from the potassium chlorate.

Asbestos paper; candle on long stick; $KClO_3$; P.

7. Use of potassium chlorate in colored fires (Bengal fires). — In all mixtures containing potassium chlorate it is of the utmost importance that each substance should be reduced to a fine powder before mixing, and that the mixing should be done on paper with the fingers (never in a mortar!!!), causing as little friction as possible. With reasonable care no difficulty will be experienced.

The ignition of colored fires is most satisfactorily effected by igniting a piece of touch-paper inserted in the top of the conical pile of powder. Ignition from a match is exceedingly uncertain and at times dangerous.

A characteristic fire may be prepared by mixing 2 g. of copper sulphate, 2.5 g. of sulphur flowers, and 15 g. of potassium chlorate. This mixture, when ignited, burns with a purple flame.

Asbestos paper; touch-paper; $CuSO_4$; $KClO_3$; S flowers.

AMMONIUM COMPOUNDS

1. Preparation of ammonium amalgam. — This interesting amalgam is prepared by the action of sodium amalgam of a soft, waxy consistency on a warm saturated solution of ammonium chloride. The sodium amalgam, when kneaded with the fingers in the ammonium chloride solution, which it rapidly attacks, swells up and forms a butterlike mass, which is porous and rapidly decomposes.

Sodium amalgam ; saturated NH_4Cl solution.

2. Preparation of ammonium amalgam by the electrolysis of a solution of ammonium sulphate. — By the electrolysis of ammonium sulphate free ammonium is liberated at the negative pole, and if an electrode of mercury is used, the ammonium amalgamates with it, forming ammonium amalgam.

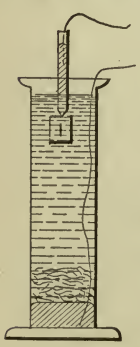

Fig. 150

A saturated solution of ammonium sulphate is placed in a 150 cc. cylinder having a 2 cm. layer of mercury on the bottom (Fig. 150). A well-insulated copper wire is thrust into the solution and the bare end pressed under the mercury. The positive electrode should consist of a small piece of platinum. On passing a current from 4 cells of a "bichromate" battery through the solution the decomposition is effected. The mercury at the bottom of the cylinder swells up with

the formation of ammonium amalgam, and care should be taken to stop the current before the mercury has reached the platinum electrode. A piece of ordinary annunciator wire which is well covered with paraffin will serve as a conductor to the mercury electrode.

Battery ; 150 cc. cylinder; annunciator wire ; Pt electrode ; Hg ; $(NH_4)_2SO_4$ saturated solution.

3. Union of ammonia and hydrochloric acid to form ammonium chloride. — (*a*) A liter cylinder is filled with hydrochloric acid gas either by downward displacement or by shaking 5 cc. of concentrated hydrochloric acid about in it for a few minutes, and is then covered with a glass plate. A brush such as is used for cleaning lamp chimneys is drenched with strongest ammonium hydroxide, and plunged suddenly into the jar of hydrochloric acid. A piece of cardboard large enough to cover the jar may be slipped over the brush handle and the brush suspended in the middle of the jar, the glass plate being quickly removed at the moment of inserting the brush. The cardboard prevents any escape of ammonium chloride fumes. As the strongest ammonium hydroxide is disagreeable to use in an open room, it is advisable to crowd the brush down into a cylinder just large enough to hold it before pouring on the ammonium hydroxide. A small quantity of ammonium hydroxide will thereby suffice to drench the brush thoroughly.

Brush ; cardboard ; con. HCl, or HCl generator ; con. NH_4OH.

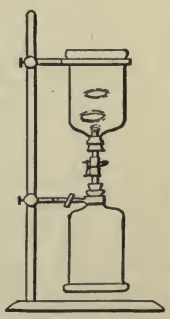

Fig. 151

(*b*) Two tubulated bell-jars, each fitted with a one-holed rubber stopper carrying a short piece of glass tubing, are connected by a piece of rubber tubing slipped over the glass tubes (Fig. 151). The

bell-jars are clamped in a vertical position and the rubber tube closed with a pinch-cock. The lower bell-jar is filled with ammonia by displacement and the upper with hydrochloric acid gas by displacement. On opening the pinch-cock the ammonia, by reason of its smaller specific gravity, ascends through the rubber tube and forms white clouds in the upper bell-jar.

Two tubulated bell-jars; corks; tubes and pinch-cock; NH_3 supply; HCl gas supply.

4. Union of ammonia and hydrogen sulphide. — Crystallized anhydrous ammonium sulphide results from the interaction of dry ammonia gas and dry hydrogen sulphide.

A clean, dry, 3 l. flask is filled with ammonia gas by displacement. When completely filled a current of hydrogen sulphide, dried by passing over calcium chloride, is conducted into the flask. On cooling the flask somewhat, a deposit of fine crystals of ammonium sulphide will appear on the interior.

$$2\,NH_3 + H_2S = (NH_4)_2S.$$

3 l. flask; NH_3 supply; H_2S supply.

5. Dissociation of ammonium sulphate solution. — Two grams of ammonium sulphate are dissolved in 300 cc. of water in a 500 cc. retort. Blue litmus solution is introduced, one drop of ammonium hydroxide being added, if necessary, to give a decidedly alkaline color. The retort is placed on a ring-stand and clamped, with the neck dipping into a 500 cc. flask containing 100 cc. of litmus solution colored red by the addition of one drop of dilute sulphuric acid. On gently heating to the boiling point (care being taken to prevent too violent ebullition, as otherwise some of the fluid made alkaline is liable to be mechanically carried over), ammonia gas distils over, and soon the upper

portion of the liquid in the receiver will be seen to have acquired a blue color, while the liquid in the retort gradually turns a decided red. The complete change in color is noticed after boiling for thirty minutes.

500 cc. retort ; 500 cc. flask ; litmus solution ; 2 g. $(NH_4)_2SO_4$.

6. Decomposition of ammonium dichromate by heat. — Ammonium dichromate, when heated, undergoes a complete decomposition, resulting in the formation of nitrogen, water, and chromium sesquioxide.

One or two grams of powdered ammonium dichromate are heated in a hard-glass test-tube till the decomposition, which is characterized by an increase in volume of the mass and an appearance of fire, begins. The heat of the decomposition, when once started, is sufficient for completion, and the mass expands, filling the tube with a tealike substance, chromic oxide. Nitrogen gas is given off, and a lighted match thrust in the tube is extinguished. At the end of the reaction the water-vapor formed escapes as steam at the mouth of the tube. The residue of chromic oxide formed may be poured upon a white plate, where its peculiar form is more easily observed.

$$(NH_4)_2Cr_2O_7 = Cr_2O_3 + 4\ H_2O + N_2.$$

Hard-glass test-tube ; white plate ; $(NH_4)_2Cr_2O_7$.

7. Decomposition of solid ammonium nitrate by zinc dust. — If ammonium nitrate in the solid form is mixed with zinc dust, the reduction is so vigorous as to produce a great increase in temperature, which, under certain conditions, causes the ignition of the zinc.

A mixture of 8 g. of ammonium nitrate and 1 g. of ammonium chloride is spread out in a thin layer on an iron plate. A layer of equal thickness of zinc dust is sprinkled on top of this layer. On allowing one drop of water to come

in contact with this mixture, the reaction takes place, and the heat developed is so great as to cause an ignition of the zinc dust. The ammonium nitrate should be previously dried, for, owing to its hygroscopic nature, it will retain enough moisture to cause a premature ignition of the zinc without the addition of water. In fact, the experiment may be performed by using a moist ammonium nitrate, and the addition of water dispensed with. Care should be taken, however, to prevent any danger from the premature ignition of the zinc dust, as it will probably be ignited before the mixture of the ammonium salts can be entirely covered with it.

Iron plate ; NH_4NO_3 ; NH_4Cl ; Zn dust.

8. Preparation of ammonium carbonate (carbamate ?) from ammonia and carbon dioxide. — Dry carbon dioxide, when allowed to come in contact with dry ammonia, forms ammonium carbamate, a constituent of commercial ammonium carbonate.

A dry 3 l. flask (Fig. 92, p. 222) is filled with ammonia by displacement, and a slow stream of dry carbon dioxide conducted into it. After a short time the walls of the flask become covered with a crystalline deposit of ammonium carbamate.

3 l. flask ; NH_3 supply ; CO_2 generator.

9. Preparation of ammonium carbonate by the interaction of ammonia and carbon dioxide in an alcoholic solution. — When carbon dioxide is conducted into an alcoholic solution of ammonia, a white crystalline deposit of ammonium carbonate (ammonium carbamate ?) is formed.

Fifty cubic centimeters of alcohol are saturated with ammonia gas and a rapid stream of carbon dioxide conducted through the solution in a beaker.

Alcohol ; CO_2 generator ; NH_3 supply.

CALCIUM

1. Action of calcium oxide with water. — Calcium oxide unites with water, with the liberation of great heat, to form calcium hydroxide.

A piece of freshly burned quicklime is immersed in water for two or three seconds and then placed on a plate. In a few moments the lime becomes very much heated, swells up, and falls to a powder.

Four or five lumps of quicklime may be placed in a large evaporating-dish and moistened with hot water. The lime swells up, filling the evaporating-dish, and giving rise to intense heat. A match may be ignited by placing the head in one of the crevices formed as the lime crumbles.

If an especially good piece of lime is available, the heat is often sufficient to ignite gunpowder. The lime should be placed on a plate and drenched with boiling water. As soon as the reaction begins, .5 g. or less of gunpowder should be sifted from a piece of paper or card upon the lime. Care should be taken to close the powder-container and remove it from any possible danger of accidental explosion before pouring the water upon the quicklime.

Crockery plates ; hot water ; fresh quicklime ; gunpowder.

2. Calcium oxide as a drying agent. — The affinity of calcium oxide for moisture makes this compound an effective drying agent for many gases.

The absorption of moisture from a gas by calcium oxide may be shown by moistening the interior of a bell-jar by holding it over a hydrogen flame and then placing it on a porcelain plate in the centre of which are a few lumps of good quicklime. A second bell-jar may advantageously be moistened and placed over a plate containing no lime. In a short time all the moisture on the walls of the bell-jar covering the lime will disappear, while the other jar will remain unchanged (Fig. 76, p. 175).

Two l. bell-jars ; 2 plates ; H flame ; fresh quicklime.

3. Preparation of calcium phosphide. — Phosphorus, when in contact with heated quicklime, forms calcium phosphide.

Five grams of well-dried yellow phosphorus are inserted in a 50 cm. length of combustion tubing, 1.5 cm. internal

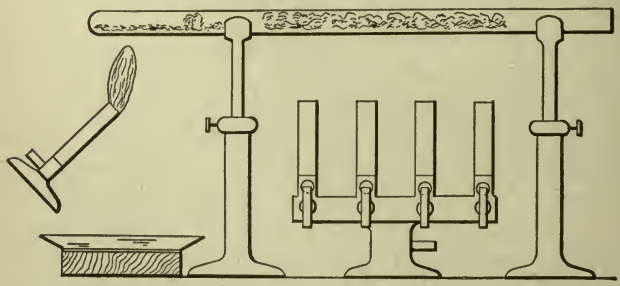

Fig. 152

diameter, closed at one end. A plug of asbestos is then introduced and a 20 cm. length of the tube filled with lumps of good quicklime (Fig. 152). The tube is so placed over a four-tube burner that the lime may be strongly heated before heating the phosphorus. When the lime has become hot, the phosphorus is brought to a boil and the vapor passed over

the heated lime. The phosphorus end of the tube should be heated with a Bunsen burner held in the hand, and a plate of sand should be placed beneath the boiling phosphorus. As the phosphorus vapor comes in contact with the hot lime, the lime glows and turns black, forming calcium phosphide.

The decomposition of the product by water is shown in Exs. 29, 30, p. 252.

50 cm. length combustion-tubing; plate of sand; 4-tube burner; fresh quicklime; P.

4. Hydration of anhydrous calcium sulphate. — When anhydrous calcium sulphate (plaster of Paris) is mixed with water, the water combines with the salt, forming a hard mass which may be used to take impressions.

A coin may be placed in the bottom of a small pill-box and the box filled with a thick, freshly prepared paste of plaster of Paris and water. On allowing the mixture to stand for half an hour it will harden. The bottom of the box can be cut away and the coin removed, leaving an impression in the plaster.

The absorption of water by plaster of Paris may be shown by simply mixing the plaster and water to a thick paste. In a few moments the mixture will have set sufficiently to remain in the beaker when it is inverted.

Pill-box; plaster of Paris.

5. Decomposition of calcium carbonate in an atmosphere of air or hydrogen. — While calcium carbonate is not decomposed in an atmosphere of carbon dioxide (see Ex. 22, p. 304), it is readily decomposed when heated in an atmosphere of hydrogen or air. (Burning limestone.)

A few lumps of calcite are placed in the bulb-tube through which a current of hydrogen freed from carbon dioxide is being passed. The issuing gas is conducted through a glass

elbow dipping into a beaker of lime-water. Until the calcite is heated the lime-water remains clear. As soon as the bulb is strongly heated, carbon dioxide is evolved and is carried by the hydrogen current into the lime-water, where it produces a precipitate.

On cooling and leaching out the contents of the bulb with water, they will be found to be strongly alkaline on account of the formation and the subsequent hydration of the calcium oxide.

The experiment may be repeated with the same results by using air freed from carbon dioxide in place of hydrogen.

$$CaCO_3 = CaO + CO_2.$$

Bulb-tube; H generator; lime-water; calcite.

STRONTIUM AND BARIUM

1. Deflagration of strontium nitrate on charcoal. — A crystal of strontium nitrate, when thrown on glowing charcoal, deflagrates vigorously, giving rise to a red flame.

If charcoal powder is heated to glowing in an iron saucer and powdered strontium nitrate is thrown into the dish, the combustion is brilliant.

Iron dish; charcoal; $Sr(NO_3)_2$.

2. Use of strontium nitrate in red fire. — Strontium nitrate is the essential coloring ingredient in red fire.

A mixture of 1 g. of potassium chlorate, 11 g. of strontium nitrate, 4 g. of sulphur flowers, and .5 g. of lamp-black, all of which have been previously finely powdered and carefully mixed on paper, burns with an intense red flame. The mixture, which should be placed in the hood, is ignited with a short piece of touch-paper.

Asbestos paper; $Sr(NO_3)_2$; $KClO_3$; S flowers; lamp-black; touch-paper.

3. Preparation of barium peroxide. — When barium oxide is heated in the presence of oxygen, it absorbs oxygen, forming barium peroxide.

A 20 cm. length of combustion-tubing provided with a one-holed rubber stopper at each end is three-fourths filled with barium oxide and so supported that it can be heated with a four-tube burner (Fig. 153). A current of oxygen,

which is allowed to bubble through sulphuric acid in a gas washing-bottle, is passed through the tube, the issuing gas bubbling through a second gas washing-bottle at the other

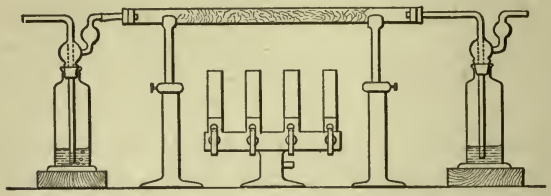

FIG. 153

end. On heating the barium oxide oxygen will be absorbed and the rate of bubbling in the second bottle will be much less than that in the first.

The barium oxide should not be heated too much, as otherwise the oxygen will again be expelled.

$$2\ BaO + O_2 = 2\ BaO_2.$$

Two gas washing-bottles; 20 cm. length combustion-tubing; 4-tube burner; O supply; BaO.

4. Decomposition of barium peroxide by heat. — A portion of the barium peroxide from the preceding experiment may be heated in a test-tube and the liberated oxygen tested with a glowing splinter.

Hydrated barium peroxide on heating yields large quantities of water, which are likely to condense and break the tube. If, however, it is carefully heated until no more steam escapes, the temperature may be raised and the oxygen liberated. The residue on treatment with water yields an alkaline solution of barium hydroxide.

5. Action of hydrogen on barium peroxide. — When barium peroxide is heated in an atmosphere of hydrogen, the reac-

tion is very vigorous, the barium peroxide becoming heated to incandescence.

A small quantity of barium peroxide is placed in a bulb-tube through which a current of hydrogen is being passed. On heating the barium peroxide slight explosions of oxy-hydrogen gas take place in the bulb, the barium peroxide appearing brilliantly incandescent.

Bulb-tube; H generator; BaO_2.

6. Barium sulphate from barium oxide and sulphur trioxide. — Sulphur trioxide unites directly with barium oxide to form barium sulphate. The evolution of heat in the operation is great and consequently the sulphur trioxide (or fuming sulphuric acid) should be placed in a test-tube clamped over a dish of sand. On carefully sifting a small quantity of barium oxide into 2 g. of the sulphur trioxide, the reaction takes place accompanied by light.

$$BaO + SO_3 = BaSO_4.$$

Plate of sand; SO_3 or fuming H_2SO_4; BaO.

7. Insolubility of barium sulphate in water. — The great insolubility of barium sulphate in water may be interestingly shown by adding sulphuric acid to successive quantities of increasingly diluted solutions of barium chloride. Fifty cubic centimeters of a saturated solution of barium chloride are placed in a 100 cc. cylinder. Five cubic centimeters of this solution are placed in a second 100 cc. cylinder and sufficient water added to dilute the solution to 50 cc. Five cubic centimeters of this dilute solution are placed in a third cylinder and diluted to 50 cc., the operation being carried out to the fifth dilution. On adding equal quantities of dilute sulphuric acid to each of the cylinders various degrees of turbidity will be obtained, the fifth cylin-

der containing, however, a distinct precipitate of barium sulphate.

This experiment may be reversed by using concentrated sulphuric acid and diluting, adding a solution of barium chloride as the reagent.

$$BaCl_2 + H_2SO_4 = BaSO_4 + 2\ HCl.$$

Five 100 cc. cylinders ; $BaCl_2$ solution.

8. Deflagration of barium nitrate and chlorate on charcoal. — A piece of charcoal having a small hollow scooped out on one side is heated to glowing in the Bunsen flame. On throwing a few crystals of barium nitrate on the charcoal the salt deflagrates vigorously.

If barium chlorate instead of barium nitrate is used, a most violent combustion takes place.

Charcoal ; $Ba(NO_3)_2$; $Ba(ClO_3)_2$.

9. Use of barium nitrate in green fire. — Barium nitrate is the essential coloring ingredient in green fire.

A mixture of 3 g. of finely powdered potassium chlorate, 8 g. of finely powdered barium nitrate, and 3 g. of sulphur flowers, when intimately mixed and ignited on asbestos paper, burns with an intense green flame.

Asbestos paper ; touch-paper ; $KClO_3$; $Ba(NO_3)_2$; S flowers.

10. Deflagration of barium chlorate. — Barium chlorate, when heated on a platinum wire in a Bunsen burner, deflagrates, imparting an intense green color to the Bunsen flame.

The deflagration may be carried out on a much larger scale by directing the Bunsen burner on a small heap of barium chlorate on asbestos paper.

When thrown on hot charcoal, the salt deflagrates vigorously.

Asbestos paper ; Pt wire ; $Ba(ClO_3)_2$.

MAGNESIUM

1. Combustion in water vapor. — The decomposition of water by magnesium, even when in a finely divided state, is at best very slow, and the hydrogen is liberated in such a manner as to prevent its collection.

At a high temperature, however, the reaction between magnesium and water vapor is intense, large quantities of hydrogen being liberated.

Three hundred cubic centimeters of distilled water are vigorously boiled in a Jena glass Erlenmeyer liter flask with a wide neck, and a lighted taper is introduced into the neck of the flask to demonstrate that water vapor is a non-supporter of the combustion of wood and to indicate that all the air is driven out of the flask. A small quantity of magnesium powder is ignited with a match on a deflagrating-spoon designed to be turned over and thus spill its contents (Fig. 154). A crude, though thoroughly practical and satisfactory spoon may be made by fastening to a stout iron wire about 30 cm. long a round piece of cork, 1 cm. thick and 2.5 cm. in diameter, on which rests a small porcelain crucible cover,

Fig. 154

whose ring is pressed into a small slit in the cork thus making it secure. The iron wire is thrust into the edge of the cork and bent upright close to it to permit of lowering the spoon into the flask. A piece of string 30 cm. long is fastened to a tack or pin in the rim of the cork 90° from the point where the iron wire is inserted. By pulling this string, if the cork is not fastened too tightly to the iron wire, the spoon may be turned through an angle of 90° and its contents spilled out.

After ignition in the air the magnesium powder is lowered one-third of the way into the flask and the string pulled, thereby allowing the powder to fall through the steam. A blinding flash equalled only by the burning of the powder in air follows.

If the string catches fire in the air, it is instantly extinguished on lowering the spoon into the flask.

Inasmuch as the combustion is very rapid it is necessary to cover the hand holding the spoon with a gauntlet.

$$Mg + H_2O = MgO + H_2.$$

Apparatus (Fig. 154); 1 l. Jena glass Erlenmeyer flask; deflagrating-spoon of special construction; gauntlets; Mg powder.

2. Combustion of magnesium in carbon dioxide. — A 300 cc. cylinder containing a layer of sand and a carbon dioxide delivery-tube extending to the bottom is filled with carbon dioxide and the dry gas allowed to flow continuously into it. Twenty-five hundredths of a gram of finely powdered magnesium (the finer the powder, the better) is placed on a 2 cm. disk of previously ignited asbestos paper, which can be easily cut out of sheet asbestos by means of a large-sized cork borer. The asbestos disk and the magnesium powder are laid on a loop in a stout iron wire thereby forming a deflagrating-spoon. On igniting the powder and quickly

lowering it into the jar till the spoon touches the sand a marked increase in the intensity of the combustion takes place. A piece of asbestos cardboard with a slit in it wide enough to hold the glass tube is quickly slipped over the top of the jar. In this way air currents formed by the combustion of the magnesium will be prevented, and thus no considerable amount of air admitted. The glass tube through which the carbon dioxide is passing is now held directly over the burning magnesium, and a lighted match inserted in the slit of the asbestos. If there is a sufficient supply of carbon dioxide, the match should be extinguished.

After the glow has died out the spoon is withdrawn and the rather compact mass of magnesium oxide and carbon is transferred by means of a spatula from the asbestos disk to a white plate. While the exterior of the mass appears white from the coating of magnesium oxide, on cutting the lump in two the interior will be seen to be perfectly black, the carbon completely covering the color of the magnesium oxide.

300 cc. cylinder; asbestos deflagrating-spoon; white plate; dry CO_2 supply.

3. Magnesium and nitric acid. — Metallic magnesium reacts with nitric acid, forming magnesium nitrate with the liberation of heat and light.

Five cubic centimeters of fuming nitric acid are gently warmed in a wide-mouthed flask. On removing the lamp and dropping .25 g. of fine magnesium powder on the hot acid, the metal takes fire, burning with almost explosive violence. (Use gauntlets and glass shields.)

The great quantity of oxides of nitrogen evolved make it necessary as a rule to perform this experiment in a hood or draft. In case the first lot of powder does not catch fire, a like quantity must immediately be poured into the neck of

the flask, gently tapping the paper in order not to drop the whole at once.

Gauntlets; shields; Mg powder; wide-mouthed 100 cc. flask; fuming HNO_3.

4. Reduction of metallic oxides and salts. — Magnesium is a very powerful reducing agent and will abstract the oxygen from many metallic oxides with explosive violence.

(*a*) *Magnesium and silver oxide.* — Equal volumes of magnesium powder and dry silver oxide are mixed on a paper and a centimeter layer poured into a small, dry test-tube. The tube is clamped to a retort-stand in such a position that a Bunsen flame may be set under it and the bottom of the tube come in the hottest part of the flame. The retort-stand is now placed between glass shields, and the lamp, resting on a plate filled with sand or on a piece of asbestos paper, is placed under the tube. (Gauntlets.) After a moment's heating, the reduction of the silver oxide takes place with an explosion. Should the tube be intact, the black metallic silver will be seen as a fine coating on the sides.

(*b*) *Magnesium and lithium carbonate.* — Lithium carbonate is energetically reduced by magnesium powder when the two are heated in equal volumes as described above.

At the moment that the reaction takes place, the fine red color of the burning lithium vapor is apparent.

$$Ag_2O + Mg = MgO + 2\,Ag.$$

$$Li_2CO_3 + Mg = MgO + CO_2 + 2\,Li.$$

Glass shields; gauntlets; Ag_2O; Li_2CO_3; Mg powder.

5. Combustion of magnesium with potassium chlorate. — A mixture of equal parts by volume of finely pulverized dry potassium chlorate and magnesium powder, when ignited on an asbestos plate, gives an instantaneous flash of blind-

ing intensity. A long taper or a touch-paper fuse should be used in igniting the mixture. Care should be taken to protect the face, owing to the great volume of smoke given off. The above mixture is commonly used in many of the so-called flashlight cartridges.

Asbestos paper ; touch-paper ; gauntlets ; colored glasses ; powdered $KClO_3$; Mg powder.

6. Formation of magnesium nitride. — At a high temperature magnesium combines with free nitrogen to form the nitride. The conditions for the formation of the maximum quantity of this compound are best secured when 1 g. of magnesium powder is heated in a porcelain crucible with a Bunsen burner. The crucible should not be more than half full of the powdered metal. The powder soon begins to burn on the surface, the product being chiefly magnesium oxide. The burning mass is gently stirred with an iron wire, and the combustion will slowly proceed for a few minutes. Under these conditions the oxygen is quickly removed from the air, and at the temperature of the combustion nitrogen is readily absorbed. The resulting product is a yellowish powder consisting of white magnesium oxide, with a varying proportion of magnesium nitride. This operation shows the method of separating atmospheric nitrogen from argon. The cooled product should be securely sealed in a bottle.

$$3\,Mg + N_2 = Mg_3N_2.$$

Porcelain crucible ; Mg powder ; iron wire.

7. Decomposition of magnesium nitride by water. — Magnesium nitride is decomposed by water, forming magnesium oxide and ammonia.

A few drops of water are allowed to fall on some of

the nitride in a test-tube. A brisk evolution of a gas is obtained, which, when tested, is seen to be ammonia.

$$Mg_3N_2 + 3\,H_2O = 3\,MgO + 2\,NH_3.$$

8. Action of hydrogen on magnesium nitride. — A current of dry hydrogen is passed through a bulb-tube containing a small quantity of magnesium nitride. On heating, ammonia will be formed and may be tested at the open end of the bulb-tube.

Bulb-tube ; H generator ; Mg_3N_2.

ZINC AND CADMIUM

1. Granulated zinc. — When melted zinc is poured in a thin stream from a height of 2 or 3 m. into a pail of cold water, the drops of melted metal spread out on striking the water, producing a form of zinc called granulated zinc. The zinc should have been previously melted in a large Hessian crucible, which is then brought into the lecture room.

FIG. 155

Instead of melting a large quantity of zinc before the lecture, the apparatus shown in Fig. 155 may be used to melt and granulate the zinc. A Hessian crucible, provided with a 3 or 4 mm. hole in the bottom, is strongly heated with a blast-lamp. Small lumps of zinc are added from time to time, and a pail of water is placed beneath the crucible. As the zinc melts, it flows out of the opening in the bottom of the crucible and drops into the water below.

Hessian crucible; ditto with 3 mm. hole in the bottom; blast-lamp; tongs; pail of water; Zn.

2. Deposition of zinc on iron (galvanized iron). — Iron that is freed from all oxide, when heated and dipped into melted zinc, becomes covered with a firmly adhering coating of the metal.

A Hessian crucible is filled with zinc, which is then melted. A large "cut" iron nail is strongly heated in the blast, and then, after cooling, carefully cleaned with hydrochloric acid and sand. After washing off all acid and dirt, the nail is again heated, dipped into a little olive or cotton-seed oil, and then plunged into the molten zinc. On removing and cooling the nail, it will be found that that portion dipped into the zinc will have become "galvanized."

Hessian crucible; tongs; cut nail; Zn; olive or cotton-seed oil.

3. Combustion of zinc dust and potassium chlorate. — Ten grams of zinc dust are mixed with 5 g. of finely powdered potassium chlorate on asbestos paper The mixture, when ignited, burns with a white flame, leaving a yellow residue of zinc oxide, which, when cold, becomes white.

Asbestos paper; Zn dust; $KClO_3$.

4. Zinc sulphide from zinc dust and sulphur flowers. — A mixture of zinc dust and sulphur flowers burns with great brilliancy, forming zinc sulphide.

Ten grams of zinc dust and 5 g. of sulphur flowers (molecular amounts) are carefully mixed on paper to prevent friction and then lighted on a piece of previously ignited asbestos paper under the hood. A brilliant greenish flame is observed. The zinc sulphide is intensely yellow while hot, but bleaches out to a considerable extent on cooling.

$$Zn + S = ZnS.$$

Asbestos paper; Zn dust; S flowers.

5. Distillation of cadmium. — Cadmium boils at 770°, and, when heated in a bulb-tube through which a current of hydrogen is being passed, the metal vaporizes and condenses as a metallic sublimate in the cooler portions of the tube.

A 4 or 5 mm. piece of metallic cadmium is placed in one arm of a bulb-tube equidistant between the bulb and the end of the arm. A current of hydrogen is then passed through the bulb, and, after all air has been expelled, the cadmium is heated until it boils. The interior of the bulb becomes covered with a metallic deposit.

Bulb-tube ; H generator ; Cd.

6. Combustion of cadmium in air. — Cadmium, at a red heat, is easily oxidized in the air.

One gram of cadmium is heated in a small porcelain crucible by means of a good strong Bunsen flame. The metal melts and soon begins to boil. The vapor ignites and gives rise to dense clouds of the brown oxide. On removing the lamp suddenly, the vapor can be seen burning in the crucible, though after a few seconds the flame goes out. The inside of the crucible will be covered with brown cadmium oxide.

A small piece of cadmium is placed in a hollow scooped out of a piece of charcoal. By directing the mouth of the blowpipe, or, better still, the blast-lamp, or oxyhydrogen jet upon it, the metal burns with more or less vividness according to the source of heat. A brown smoke of cadmium oxide rises from the burning globule.

$$2\ Cd + O_2 = 2\ CdO.$$

Porcelain crucible ; blowpipe and charcoal ; Cd.

7. Preparation of cadmium sulphide. — When hydrogen sulphide is conducted into a cold, neutral solution of cadmium chloride, a yellow precipitate of cadmium sulphide is

obtained. If, however, the cadmium solution is acidified and kept hot, the sulphide precipitated is of an orange-red color.

A current of pure hydrogen sulphide is conducted into a series of two gas washing-bottles, the first of which contains a cold, neutral solution of cadmium chloride, the second containing a hot solution of cadmium chloride acidified with hydrochloric acid. The second gas washing-bottle is placed in a beaker containing hot water. On passing hydrogen sulphide through the system, cadmium sulphide is precipitated in both bottles and is of a yellow color in the first and of an orange-red in the second. The solution in the first bottle should not contain an excessive amount of cadmium chloride, as otherwise considerable time will be required to saturate it with hydrogen sulphide.

Two gas washing-bottles; large beaker of hot water; H_2S generator; $CdCl_2$ solution.

MERCURY

1. Purification and filtration. — Mercury may be freed from much foreign material by filtering through fine linen or cambric, or through a dry, folded filter-paper having a pinhole in the bottom.

The chemical purification of mercury is effected by shaking the metal with very dilute nitric acid or dilute ferric chloride solution in a large separating-funnel. The purified metal should be well washed with water, the excess of water being removed by placing filter-papers on the surface of the metal.

Large separating-funnel; linen cloth; Hg.

2. Iron amalgam. — Under ordinary conditions iron does not amalgamate. If, however, a piece of clean sheet-iron is rubbed with liquid sodium amalgam and a filter-paper wet with saturated ammonium chloride solution, the resulting ammonium amalgam produces an amalgamation of the iron.

Sheet iron; Na amalgam; saturated NH_4Cl solution.

3. Preparation of mercurous iodide from mercury and iodine. — Mercury and iodine, when intimately rubbed in molecular proportions, form an amorphous green powder consisting of mercurous iodide.

Sixteen grams of mercury are placed in a mortar with 10 g. of iodine. A few drops of alcohol are then added,

and the mixture intimately rubbed with a pestle. In a few moments a green amorphous powder will be formed. At times certain portions of the powder will appear red, but when all the globules of mercury have disappeared, the whole mass will be green. The compound should be preserved in the mortar for use in the following experiment.

$$2\,Hg + I_2 = Hg_2I_2.$$

Mortar; Hg; I.

4. Preparation of mercuric iodide from mercurous iodide and iodine. — When mercurous iodide is rubbed in a mortar with an excess of iodine, it is converted to mercuric iodide.

Ten grams of iodine are added to the mercurous iodide in the mortar from the preceding experiment, and the mixture thoroughly rubbed with the pestle. In a short time the green mercurous iodide will be completely converted to the red mercuric iodide, all the iodine being taken up in the reaction.

$$Hg_2I_2 + I_2 = 2\,HgI_2.$$

Mortar and contents of preceding experiment; I.

5. Formation of mercuric iodide from potassium iodide and mercuric chloride. — A striking example of a dry reaction is afforded by rubbing together equal weights of finely powdered mercuric chloride and finely powdered potassium iodide in a porcelain mortar. The two powders, when first placed in the mortar, are perfectly white, but on intimately mixing them with the pestle the mixture becomes red from the formation of mercuric iodide.

Mortar and pestle; finely powdered $HgCl_2$; finely powdered KI.

6. Preparation of artificial cinnabar. — A solution of mercuric chloride is treated with ammonium hydroxide as long as any precipitate is formed. A strong solution of sodium

thiosulphate is then added to the liquid until the precipitate is completely dissolved. If the liquid thus prepared is heated for some time in a beaker of water at a temperature of 70°, a red precipitate of cinnabar is formed.

If a portion of the liquid is suddenly brought to a boil, the cinnabar is immediately precipitated, but it has a brownish red color.

Beaker of hot water; $HgCl_2$ solution; $Na_2S_2O_3$ solution.

COPPER

1. Deposition on iron. — Iron replaces copper in solutions of cupric sulphate, forming ferrous sulphate and copper.

A cleaned knife-blade immersed for a few moments in a solution of cupric sulphate becomes covered with a thin coating of metallic copper.

The complete removal of copper from solutions may be shown by placing three or four nails in a solution of cupric sulphate and allowing the vessel and its contents to stand twenty-four hours. All the copper will have been precipitated on the iron, the solution will have lost its blue color, and on the addition of ammonium hydroxide a greenish precipitate instead of a deep blue color will be obtained.

A piece of zinc when immersed in cupric sulphate solution likewise replaces the copper, and this metal may be used in place of iron in the above experiment.

$$CuSO_4 + Fe = FeSO_4 + Cu.$$

Knife; iron nails; Zn rod; $CuSO_4$ sol.

2. Electrical deposition of copper (copper plating). — When a current of electricity is passed through a cupric sulphate solution, copper is deposited on one electrode while oxygen is liberated at the other (Ex. 7, p. 13).

Two platinum electrodes are connected with a bichromate battery and then immersed in a solution of cupric sulphate.

Immediately the positive electrode becomes covered with a red coating of copper. The negative electrode remains unchanged. By reversing the current the copper is deposited on the clean electrode while the other gradually loses its coating, ultimately becoming perfectly bright and free from copper.

Cards with the plus and minus signs should be placed beside the electrodes.

Bichromate battery; 2 Pt electrodes; + and − signs; $CuSO_4$ solution.

3. Color of copper. — The color noticed on ordinary copper is partially due to the presence of small quantities of a superficial coating of the oxides of copper. The true color of copper is shown by the reduction of hot cupric oxide by alcohol vapor.

One cubic centimeter of ethyl or methyl alcohol is poured into a good-sized test-tube loosely fitted with a rubber stopper. A copper spiral (Ex. 38, p. 37) is then heated till it is thoroughly oxidized, and while still hot carefully introduced into the test-tube containing alcohol. Immediately the cupric oxide coating on the spiral is reduced, and the color of metallic copper is seen. The rubber stopper is held loosely in the test-tube until the expansion of the gases in the tube ceases. At that moment the cork is firmly introduced to prevent any air from entering the tube.

After cooling, the cork is withdrawn, and the spiral may be taken out and examined. This is essentially the method of preparing the reduced copper spiral used in elementary organic analysis.

Copper spiral (Ex. 38, p. 37); alcohol.

4. Action of light on cuprous chloride. — Sunlight acts on cuprous chloride, changing its color from white to dark violet.

A quantity of the freshly precipitated chloride may be exposed to sunlight on a porcelain plate.

On immersing a strip of polished sheet copper in a concentrated solution of cupric chloride, the copper will become covered with a coating of insoluble cuprous chloride. The copper should then be washed with running water, and a figure cut out of heavy paper placed over it. On exposure to sunlight it will be found that the uncovered portion of the sheet will become dark colored, while the covered portion will remain light.

Cu sheet; heavy paper; $CuCl_2$ solution; freshly precipitated Cu_2Cl_2.

5. Flame coloration by cuprous chloride. — The coloration of a flame by cuprous chloride may be produced by holding a piece of copper gauze in the flame and conducting a current of chlorine into the opening at the base of a Bunsen burner (Fig. 156). A coloration due to the cuprous chloride is imparted to the flame.

FIG. 156

The gauze should first be strongly heated and all shellac and foreign material burned off. Chlorine admitted at the base of the burner, one bubble at a time, produces intermittent flashes of color in the flame which become continuous with a steady introduction of chlorine.

Cu gauze; small Cl generator.

6. Solubility of cupric chloride in alcohol. — Cupric chloride dissolves readily in alcohol, yielding a colored solution which may advantageously be used to show the flame coloration produced by this salt.

A piece of previously ignited asbestos paper is saturated

with an alcoholic solution of cupric chloride. On igniting the alcohol, a green flame is obtained.

Asbestos paper previously ignited ; alcohol ; $CuCl_2$.

7. Use of cupric chloride in colored fire. — A mixture of 2 g. of powdered charcoal, 2 g. of cupric chloride, and 4 g. of potassium chlorate, ignited on asbestos paper, yields a blue flame.

Asbestos paper ; $KClO_3$; $CuCl_2$; powdered charcoal.

SILVER

1. By electrolysis. — (*a*) When an electric current is passed through a solution of potassium cyanide containing silver, the silver is deposited at one pole.

Sufficient potassium cyanide solution is added to a solution of silver nitrate to redissolve the precipitate first formed. The electrode connected with the zinc of a bichromate battery should consist of a well-cleaned and polished strip of copper. The other electrode may be a piece of platinum. On passing a feeble current through the solution, a fine deposit of silver is formed on the copper.

Bichromate battery ; Cu sheet ; Pt foil ; $AgNO_3$ solution ; KCN solution.

(*b*) By the electrolysis of a neutral solution of silver nitrate silver may be deposited in a crystalline form.

A concentrated solution of the salt is placed in a beaker, and two platinum electrodes, which are connected with two bichromate cells, are immersed in the liquid. On the negative pole silver will be deposited in a crystalline mass.

Bichromate battery ; 2 Pt electrodes ; $AgNO_3$ solution.

2. Precipitation by mercury. — A few grams of mercury are placed in a small, fine-meshed linen bag and immersed

beneath the surface of silver nitrate solution in a large beaker (Fig. 157). In the course of a day or two a fine network of crystallized silver will be found clinging to the bag, forming the so-called silver tree.

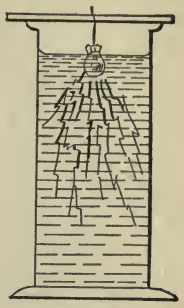

Fig. 157

Large beaker; fine-meshed linen bag; $AgNO_3$; Hg.

3. Formation of silver iodide in dilute solutions. — In a solution containing a mixture of a chloride, a bromide, and an iodide, to which a solution of silver nitrate has been added, the iodide is always first precipitated, then the others. To illustrate this, a saturated solution of sodium chloride is used, to which 5 drops of a solution containing 2 g. of potassium iodide in 50 cc. of water are added. This gives a very small amount of the iodide in the presence of a large amount of the chloride. The addition of 3 to 5 drops of silver nitrate solution, containing 2.5 g. in 50 cc. of water, produces a decidedly yellowish precipitate consisting of silver iodide. On adding the same amount of silver nitrate to a saturated solution of sodium chloride free from iodide, the color of the precipitate is a pure white. In the presence of a very large excess of a chloride, the small amount of iodide is therefore first precipitated. The slight difference in color prevents the demonstration of the analogous case of the bromide, for, while the color of silver iodide is markedly different from the color of the chloride, the same is not true of the differences in color between the bromide and the other two compounds.

Saturated NaCl solution; 2 g. of KI; 2.5 g. of $AgNO_3$.

4. Color change of silver iodide on heating. — Silver iodide, when heated, changes its color, becoming intensely yellow.

A figure or character is marked with a dilute solution of silver nitrate on a card, which is then dried out of contact with the light. The card is then flowed with a solution of potassium iodide, which converts the silver nitrate to silver iodide. After all excess of potassium iodide has been washed off, the card is carefully dried. On heating it above a Bunsen flame, the character, which was originally very indistinct, appears in bright yellow lines. On cooling, the color disappears.

Card prepared with AgI.

ALUMINIUM

1. Combustion in air. — Aluminium wire or aluminium sheet does not burn in the air. A piece of wire held in the flame melts with superficial oxidation, but does not burn. Aluminium sheet acts in a similar manner. Aluminium filings, unless very fine, do not readily burn by being strewn through a flame. Aluminium powder strewn through a flame burns with great brilliancy.

Aluminium sheet, wire, filings, leaf, powder.

2. Combustion in oxygen. — Aluminium leaf, when strongly heated in a current of oxygen, burns brilliantly to form aluminium oxide.

A bulb-tube is filled with aluminium leaf, and a small bit of string or a shred of filter-paper is introduced to serve as a kindler. On passing a current of oxygen through the bulb and strongly heating the leaf, the string catches fire and imparts the flame to the aluminium leaf, which burns in the atmosphere of oxygen with a brilliant flash.

$$4\ Al + 3\ O_2 = 2\ Al_2O_3.$$

Bulb-tube; string; O supply; Al leaf.

3. Action of sodium amalgam. — Mercury does not unite readily with aluminium to form an amalgam, though if liquid sodium amalgam is used the aluminium amalgam is readily formed. Aluminium, when amalgamated, is easily oxidized,

and in the presence of moist air aluminium oxide is rapidly formed.

A character or letter is drawn on a piece of sheet aluminium, well cleaned and free from oil, by using a clean copper wire dipped in liquid sodium amalgam. Almost immediately a mossy growth of aluminium oxide appears, rising perpendicularly from the aluminium sheet in sharply cut lines to a height of several millimeters.

Al sheet; Cu wire; Na amalgam (liquid).

4. Reduction of metallic oxides by aluminium powder. — Ferric oxide, when mixed with half its weight of aluminium powder and strongly heated in a crucible, is reduced by the aluminium with an explosion. The powders should be intimately mixed and placed in a small crucible, and a disk of asbestos paper should be pressed down upon them. As considerable heat is required to start the reaction, it may be necessary to use the blast-lamp.

Lead monoxide, when mixed with aluminium powder and heated, is likewise reduced, producing an explosion. Four grams of litharge are mixed with .25 g. of aluminium powder.

Cupric oxide in the form of a powder, when mixed with one-third its weight of aluminium powder, also explodes on ignition.

Small crucibles; asbestos paper; powdered Al, Fe_2O_3, PbO, and CuO.

5. Explosion of aluminium powder and sodium peroxide. — When aluminium powder and anhydrous sodium peroxide are mixed and moistened with a drop of water, the mixture explodes.

Sodium peroxide is placed in a clean, dry test-tube and an equal volume of aluminium powder added. The two powders are well mixed by shaking, and the test-tube is

clamped behind a glass screen. One drop of water is allowed to flow from a long glass tube into the test-tube. The resulting explosion shatters the tube.

Glass screen ; long glass tube ; Na_2O_2 ; Al powder.

6. Union of aluminium and bromine. — Aluminium and bromine while not uniting in the cold react very energetically at a higher temperature. Aluminium filings are placed in the bottom of a test-tube clamped in a vertical position. After heating until the glass is just red, 10 drops of bromine are carefully poured from a dry test-tube into the heated tube. The elements unite with a vivid combustion, yielding anhydrous aluminium bromide.

On cooling, 2 or 3 drops of water may be allowed to fall into the tube, where they react with the anhydrous bromide with a hissing sound.

$$2\ Al + 3\ Br_2 = 2\ AlBr_3.$$

Al filings ; Br.

7. Union of aluminium powder and iodine. — One-fourth of a gram of aluminium powder is heated in a thick-walled test-tube to faint redness. One-half a gram of finely powdered iodine is shaken into the tube out of a folded paper. The aluminium unites with the iodine with brilliant combustion. If the Bunsen flame is held at the mouth of the tube for a moment, the vaporized aluminium iodide will catch fire and burn. On cooling, a mass of aluminium iodide mixed with some uncombined aluminium will be seen in the bottom of the tube.

If 1 or 2 drops of water are allowed to fall on the cooled aluminium iodide in the test-tube, a vigorous action takes place, accompanied by great heat.

$$2\ Al + 3\ I_2 = 2\ AlI_3.$$

Al powder ; I.

8. Absorptive power of aluminium hydroxide for coloring matter. — Aluminium hydroxide absorbs certain coloring matters, and this property is much used in the process of dyeing.

An infusion of logwood is prepared by boiling for two minutes a few pieces of the wood with water. One hundred cubic centimeters of the colored liquid are placed in a large mortar, and one-third of its volume of a thick paste of moistened, well-washed, freshly precipitated aluminium hydroxide is added. The mixture is rubbed with a pestle for several minutes, and then washed upon a large filter. Unless a too concentrated solution of logwood has been used, the filtrate will appear perfectly colorless.

If aluminium hydroxide is precipitated from an alum solution which is somewhat colored with logwood extract, the hydroxide will combine with the coloring matter and settle as a colored precipitate, leaving the supernatant liquid clear. The alum solution should be distinctly colored, and then aluminium hydroxide added in slight excess.

Cotton fibres, when boiled with logwood extract, absorb but a very small amount of the coloring matter. If, on the other hand, the fibres are impregnated with aluminium salts, sufficient color is deposited on the fibre to make the process applicable for dyeing.

A strip of well-washed white cotton cloth is soaked in a solution of aluminium acetate, prepared by dissolving aluminium hydroxide in an insufficient quantity of acetic acid. The strip of cloth, together with a fresh strip, is immersed in logwood extract and boiled, with constant stirring for a few minutes. The cloth impregnated with the aluminium salt will acquire a deep color.

Mortar and pestle; cotton cloth; logwood chips; $Al(OH)_3$ paste; $KAl(SO_4)_3$ solution.

9. Preparation of potassium aluminium sulphate (potassium alum). — One molecule of aluminium sulphate combines with 1 molecule of potassium sulphate, forming a double sulphate which crystallizes with 24 molecules of water. Owing to its relative insolubility in water, it is readily prepared by mixing equal volumes of saturated solutions of potassium sulphate and aluminium sulphate. On shaking the test-tube, a crystalline precipitate is obtained.

A saturated solution of potassium chloride may be used, in the place of the potassium sulphate solution, with the same result.

10. Utilization of alum in clarifying water. — When a solution of alum is added to a natural water which is slightly alkaline, aluminium hydroxide is precipitated in a very finely divided condition, and, as the flocculent precipitate settles, it entangles with it any solid organic matter or sediment which may be present in the water.

A few cubic centimeters of alum solution are added to some turbid water in a 2 l. beaker, and the solution is allowed to stand over night. A second beaker should be filled with the water and no alum solution added to it. The next day it will be found that the beaker containing the alum solution in the water will be perfectly clear, all the impurities having settled to the bottom with the aluminium hydroxide. The contrast between this and the other beaker will be very marked.

Two 2 l. beakers; turbid water; alum solution.

TIN

1. Deposition by electrolytic action. — A small porous cup half filled with very dilute sulphuric acid is placed in a beaker containing concentrated stannous chloride solution. A rod of zinc, to which a copper wire is attached, is placed in the porous cup and the end of the copper wire immersed 1 cm. beneath the surface of the stannous chloride solution. The apparatus thus arranged is placed in a quiet spot and covered with a bell-jar. In the course of 24 hours, a net-work of tin crystals will be formed in the liquid surrounding the porous cup (Fig. 158).

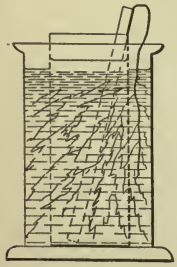

FIG. 158

500 cc. beaker; porous cup; Zn rod with Cu wire; $SnCl_2$ solution.

2. Deposition on zinc. — A solution of stannous chloride is decomposed by the action of metallic zinc, tin being deposited in the form of fine crystals.

A zinc rod is immersed in a rather concentrated solution of stannous chloride. A mossy deposit, consisting of minute crystals of tin, forms about the zinc.

That the mossy vegetation obtained by immersing a strip of zinc in tin chloride solution consists of minute crystals of the metal, is shown by firmly pressing a quantity of the

spongy mass between the fingers until all water is removed. The crystals knit together so closely that the mass appears as a solid lump, which may be polished by rubbing with a piece of chamois skin.

By using a more dilute solution of the chloride and allowing the reaction to take more time, larger crystals of tin may be obtained, and a treelike deposit will be formed.

$$SnCl_2 + Zn = Sn + ZnCl_2$$

$SnCl_2$; Zn rod.

3. Crystalline structure. — On bending a rod of tin or a piece of tin pipe, the crystals rub on each other producing a peculiar crackling sound called the "tin cry."

When molten tin is allowed to cool, it assumes a crystalline structure.

A piece of common tinned iron is heated over a Bunsen burner until the tin begins to be discolored. It is then thrust quickly into cold water and the surface rubbed with a piece of filter-paper moistened with dilute aqua regia. After removing the excess of acid by rubbing with a little dilute sodium hydroxide solution, the surface of the metal will be found covered with well-marked crystalline figures.

Block tin rod or pipe; sheet tinned iron.

4. Preparation of stannic chloride. — Chlorine when brought in contact with heated tin unites with it, forming stannic chloride.

A few pieces of granulated tin are placed in the larger distilling flask shown in Fig. 159. A current of dry chlorine is conducted through the glass elbow in the neck of the flask and caused to play upon the surface of the tin. The side tube is connected with a small distilling flask immersed

in ice-water. The excess of chlorine, together with any uncondensed vapors of stannic chloride, is conducted to a flue.

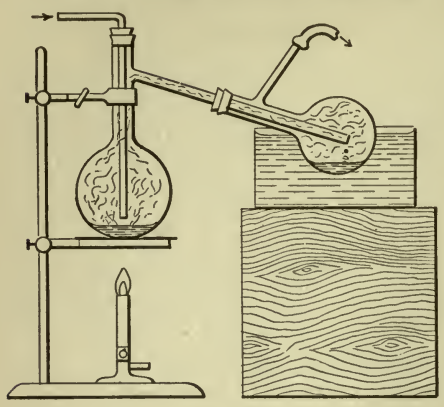

Fig. 159

The tin is carefully heated till it melts, when it will be seen that the reaction with the chlorine has begun as the flask fills with dense white fumes. The chloride condenses for the most part in the small flask to a light-colored, fuming liquid.

$$Sn + 2\ Cl_2 = SnCl_4.$$

Apparatus (Fig. 159); Cl generator; Sn; ice.

5. Action of stannic chloride in the air. — Stannic chloride fumes strongly in the air and rapidly absorbs moisture, forming a crystalline hydrate.

One cubic centimeter of stannic chloride is poured into a dry 400 cc. crystallizing-dish, and a piece of filter-paper placed over it. The chloride soon absorbs moisture from the air, and forms a solid crystalline mass on the bottom and sides of the dish.

Much larger crystals are obtained by pouring 1 cc. of the chloride into a 500 cc. flask, which is allowed to stand over night. The stannic chloride will slowly evaporate and, rising through the narrow neck, come in contact with the moisture of the air and form a network of crystals across

the mouth of the flask. The flask is filled with the fumes of stannic chloride, and by blowing moist air through a glass tube into the flask dense white clouds are formed.

400 cc. crystallizing-dish ; 500 cc. flask ; $SnCl_4$.

6. Stannous iodide from stannous chloride and potassium iodide. — When concentrated solutions of stannous chloride and potassium iodide are mixed, a yellow crystalline precipitate of stannous iodide appears suddenly in the solution.

$$SnCl_2 + 2\ KI = SnI_2 + 2\ KCl.$$

Concentrated solutions of $SnCl_2$ and KI.

7. Preparation of stannic sulphide (mosaic gold). — A mixture of equal parts of powdered tin and sulphur flowers is mixed with one-eighth of its volume of powdered ammonium chloride. The mixture is then placed in a porcelain crucible, covered with a 2 mm. layer of powdered ammonium chloride, and the covered crucible heated with a Bunsen burner. As a result of the reaction, the ammonium chloride vaporizes as a white smoke, and stannic sulphide sublimes as a brilliant yellow crystalline deposit on the crucible lid.

Porcelain crucible ; Sn ; S flowers ; NH_4Cl.

LEAD

1. Preparation of pyrophoric lead. — Finely divided lead ignites spontaneously when exposed to the air. By the ignition of lead tartrate, a mixture of finely divided lead and carbon is obtained.

Five grams of lead tartrate are gently heated in an ignition tube until the evolution of gas ceases. It is important that the heat should not be carried to too high a degree, as the small particles of lead are liable, under those circumstances, to fuse together partially, and thus spoil the experiment. As soon as the gases cease coming off, the tube is corked with a well-fitting rubber stopper to insure an air-tight closure. After the tube has become perfectly cold, the cork may be removed and the finely divided lead poured out upon a plate, best from a height of half a meter. On exposure to the air, the lead catches fire spontaneously.

Preparation of lead tartrate. — Lead acetate solution is added to a solution of tartaric acid till there is no more precipitate formed. The precipitate, which is quite insoluble in water, may be filtered off, washed with water, and dried in an air-bath. It loses its crystal water at 130°, and in this form is best suited for the above experiment, since the water is likely to condense and break the tube.

Hard-glass test-tube with well-fitting rubber stopper ; white plate ; lead tartrate.

2. Deposition on zinc (lead tree). — The deposition of lead from a solution of lead nitrate upon a zinc rod is obtained by covering a rod of zinc, such as is used in batteries, with one layer of asbestos paper. The rod is then suspended in a moderately strong (10 per cent) solution of lead nitrate, and allowed to stand till the next exercise. Crystals of lead will appear on the outside of the rod (Fig. 160).

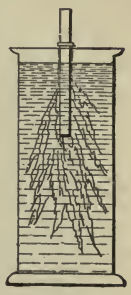

FIG. 160

A similar rod, not covered with paper, when dipped into a solution of lead nitrate, is immediately covered with a black, mossy deposit of finely divided lead.

$$Pb(NO_3)_2 + Zn = Zn(NO_3)_2 + Pb.$$

Asbestos paper; $Pb(NO_3)_2$ solution (10 per cent); Zn rods.

3. Crystalline structure of lead. — If a piece of sheet-lead is brushed over with strong nitric acid, a crystalline structure will be developed. The excess of acid should be removed by washing with water.

As the lead oxidizes rapidly, the figures soon disappear.

Sheet-lead; concentrated HNO_3.

4. Action of water on lead. — A large strip of clean, bright lead is suspended in a beaker of distilled water for several hours. A white precipitate is formed, part of which settles to the bottom, while quite a large quantity coats the lead, giving it a corroded appearance.

Hydrogen sulphide added to the water produces a black precipitate of lead sulphide.

By dissolving slight quantities of certain salts in water in different beakers, and allowing the lead to remain in them, a very striking comparison may be made of the respective

merits of potable waters containing those salts when used with lead pipes.

The above described experiment is repeated, using a beaker containing a liter of water, in which .30 g. of dry potassium carbonate and .04 g. of potassium nitrate have been dissolved. After standing 24 hours, the solution is tested with hydrogen sulphide. While in the beaker of pure distilled water an intense black is formed, the water containing the salts gives no reaction with hydrogen sulphide, since the salts entirely prevent any solvent action of the water.

2 large beakers; 2 large pieces of sheet Pb; distilled water; K_2CO_3; KNO_3.

5. Oxidizing action of lead peroxide. — (*a*) *On sulphur.* — When dry, freshly prepared lead peroxide is rubbed in a mortar with a small bit of sulphur, the mixture takes fire.

Gauntlets; mortar and pestle; PbO_2; S flowers.

(*b*) *On hydrogen sulphide.* — A stream of hydrogen sulphide is allowed to impinge on a gram or two of dry, freshly prepared lead peroxide. On coming in contact with the oxide, the gas is oxidized, and bursts into a flame. The surface of the lead peroxide soon turns a lighter color, owing to the formation of lead sulphate. By stirring the powder, and thereby exposing fresh surfaces to the action of the gas, nearly the whole mass may ultimately be converted to lead sulphate.

The powder may be allowed to fall by the end of a tube through which hydrogen sulphide is issuing. The gas will be ignited.

H_2S generator; PbO_2.

(*c*) *On nitrobenzene.* — The strong oxidizing action of lead dioxide may be shown by heating 1 g. of the substance

with 2 or 3 drops of nitrobenzene in a test-tube clamped behind glass screens. On heating the tube an explosion occurs.

Glass screens; PbO_2; nitrobenzene.

6. Explosion of lead nitrate and sulphur. — Lead nitrate, when rubbed with sulphur, oxidizes it with explosive violence.

Equal quantities of finely pulverized lead nitrate and sulphur flowers should be placed in an unglazed mortar, and rubbed with the pestle in the gloved hand. Very small quantities of the mixture should be used.

Mortar and pestle; gauntlets; $Pb(NO_3)_2$; S flowers.

BISMUTH

1. Fusion and crystallization. — Bismuth melts at a comparatively low temperature and, on cooling, crystallizes in rhombohedrons which appear almost cubical. The crystallization is much facilitated by heating the metal for some time with a small quantity of potassium nitrate. The fusion is made in a porcelain crucible or a Jena glass beaker. As soon as the mass has cooled sufficiently to form a crust on the surface, a hole is made in the crust and the molten metal poured out. On breaking open the shell, the cavity will be found to be lined with crystals.

The play of colors observed in heating and cooling copper (Ex. 5, p. 332) is also observed in heating bismuth.

Porcelain crucible or Jena glass beaker; Bi; KNO_3.

2. Fusible alloys. — Tin and bismuth form ingredients of all of the fusible alloys, the most interesting of which is the so-called Wood's metal, having a melting point of about 70° C., and consequently melting in hot water.

This alloy is made by melting together 15 g. of cadmium, 20 g. of tin, 40 g. of lead, and 80 g. of bismuth.

Other alloys formed by varying these proportions are Rose's metal, melting at 93°.5, and Newton's metal, melting at 94°.5.

Wood's metal; Rose's metal; Newton's metal; Pb; Bi; Cd; Sn.

CHROMIUM

1. Preparation of anhydrous chromium chloride. — A mixture of equal volumes of powdered anhydrous chromium oxide and charcoal powder is placed in a bulb-tube through which a current of chlorine is being passed. On heating the mixture strongly with a large flame, a violet crystalline deposit of chromium chloride is obtained in the colder portions of the tube.

The anhydrous salt is insoluble in water.

$$Cr_2O_3 + 3\ C + 3\ Cl_2 = 2\ CrCl_3 + 3\ CO.$$

Bulb-tube; Cl generator; Cr_2O_3; charcoal powder.

2. Preparation of chromium trioxide (chromic acid). — Sixty cubic centimeters of concentrated sulphuric acid are slowly poured into 40 cc. of a cold saturated solution of potassium dichromate. The mixture is then cooled, and the resulting crystalline precipitate, consisting of chromium trioxide, filtered on a funnel containing glass wool or fibrous asbestos.

$$K_2Cr_2O_7 + H_2SO_4 = K_2SO_4 + 2\ CrO_3 + H_2O.$$

Funnel; glass wool; cold saturated solution of $K_2Cr_2O_7$.

3. Oxidizing action of chromium trioxide. — (*a*) *On alcohol.* — If absolute alcohol is allowed to drop upon a few

crystals of dry chromium trioxide, the alcohol is oxidized and ignited.

CrO_3 crystals; absolute alcohol.

(*b*) *On wood.* — Chromic acid, or potassium dichromate in the presence of sulphuric acid, oxidizes wood to carbon dioxide even in the cold. A splinter of wood, when thrust into a warm solution of potassium dichromate and sulphuric acid, is instantly colored brown, and small quantities of a gas are evolved.

Sawdust, when mixed with a solution of potassium dichromate and sulphuric acid, is rapidly oxidized to carbon dioxide with the liberation of great heat. The mixture is made in a 500 cc. flask, fitted with a one-holed cork, and a delivery-tube dipping into lime-water. In a few moments, even without the application of external heat, a reaction begins, large quantities of carbon dioxide are evolved, and the mixture becomes very hot. The battery solution described on p. 3 may be used in these experiments with success.

Splinter; sawdust; $K_2Cr_2O_7$ or battery solution; lime-water.

4. Dyeing with chrome yellow. — By precipitating chrome yellow on the fibres of cloth, the color is sufficiently retained to dye the fabric.

A strip of well-washed cotton cloth is dipped in a solution of lead acetate and then immersed in a solution of potassium dichromate. The cloth becomes covered with a bright yellow, finely divided precipitate of lead chromate.

Cotton cloth; solutions of $K_2Cr_2O_7$ and $Pb(C_2H_3O_2)_2$.

5. Oxidizing action of lead chromate. — Lead chromate readily gives up its oxygen when heated with organic matter, and is much used as an oxidizing agent in elementary organic analysis.

A small quantity of pulverized sugar is mixed with three or four times its volume of lead chromate, and the mixture is heated in a test-tube. The carbon dioxide may be conducted through a glass tube into lime-water, where it will produce a white precipitate.

Test-tube with cork and delivery-tube; sugar; $PbCrO_4$ fused and powdered; lime-water.

6. Preparation of chromium oxychloride (chromyl chloride). — When a mixture of sodium chloride and potassium chromate is heated with concentrated sulphuric acid, a dark red liquid, chromium oxychloride, distils over.

Ten grams of fused, powdered sodium chloride should be heated to fusion in a crucible with 17 g. of potassium chromate. The cooled mass is then pulverized and placed in a 500 cc. glass-stoppered retort, the neck of which is thrust into a filter flask used as a receiver (Fig. 93, p. 223). Thirty grams of fuming sulphuric acid or 25 g. of concentrated sulphuric acid, mixed with 5 g. of sulphur trioxide, are poured through a long-stemmed funnel or thistle-tube upon the mixture of the salts. When the retort is gently warmed, dense red fumes, which distil over and condense in the receiver to a dark-colored liquid, appear.

The oxidizing action of chromium oxychloride may be shown by dropping a 1 or 2 mm. piece of well-dried phosphorus into 2 or 3 drops of the liquid placed in the test-tube shown in Fig. 109, p. 262. As the phosphorus comes in contact with the liquid, an explosion is obtained.

A current of hydrogen sulphide, directed upon a small quantity of the liquid in an evaporating dish, is immediately oxidized and ignited.

Ammonia gas likewise, when directed upon the liquid, reacts with evolution of dense fumes.

A filter paper moistened with alcohol is touched with a drop of liquid. The alcohol is ignited.

$$K_2CrO_4 + 2\,NaCl + 2\,H_2SO_4 = Na_2SO_4 + K_2SO_4 + CrO_2Cl_2 + 2\,H_2O.$$

500 cc. glass-stoppered retort; filter flask; apparatus (Fig. 109, p. 262); H_2S generator; NH_3 generator; fused, powdered NaCl; K_2CrO_4; alcohol; P.

IRON

1. Reduction of ferric oxide by hydrogen. — Ferric oxide is reduced to metallic iron when heated in a current of hydrogen. If the temperature at which the reduction is carried out is not too high, the reduced iron is pyrophoric.

A thin layer of ferric oxide is placed in a 20 cm. length of combustion-tubing fitted at each end with a one-holed rubber stopper and a glass tube. A current of well-washed hydrogen is passed slowly through the tube, and the iron oxide is heated to as low a temperature as will suffice to effect the reduction. When no more water escapes from the open end of the tube, the reduction is complete and the reduced iron is allowed to cool in a current of hydrogen. On shaking a small quantity of the cold powder upon a plate, it will rapidly oxidize and glow in the air.

Some of the cooled powder may be allowed to fall upon a small heap of gunpowder. The precautions described in Ex. 3, p. 330, should be carefully observed here.

Precipitated, well-washed, and dried ferric hydroxide is best used for reduction when pyrophoric iron is desired. The combustion-tube may be drawn out at both ends and sealed off after the reduction is complete. The tube thus prepared can be kept indefinitely and the pyrophoric character of the iron remain unchanged.

$$Fe_2O_3 + 3\,H_2 = 2\,Fe + 3\,H_2O.$$

20 cm. length combustion-tubing; 1-holed stoppers; H generator with gas washing-bottles; gunpowder; Fe_2O_3; $Fe(OH)_3$.

2. "Passive" iron. — When iron is immersed in fuming nitric acid, it becomes resistant to the action of acids and other reagents, being in the so-called passive state. The passivity is explained as the result of a thin, resistant coating on the iron of either iron oxide or oxides of nitrogen.

A piece of sheet iron is freed from all oil and carefully cleaned. The iron is then suspended on a fine platinum wire and is completely immersed in fuming nitric acid. As the iron enters the acid, a vigorous evolution of oxides of nitrogen is obtained. The reaction is, however, of only a few seconds' duration. The iron is then carefully withdrawn, immersed in water, and then in ordinary concentrated nitric acid. The iron is no longer acted upon by the acid.

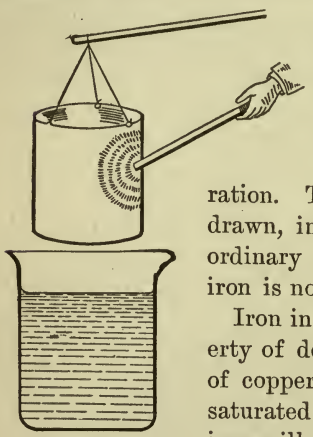

FIG. 161

Iron in the passive state loses its property of depositing copper from solutions of copper salts, and, when immersed in a saturated solution of copper nitrate, the iron will retain its original grayish color. The iron should be carefully withdrawn from the solution of copper nitrate, and then sharply struck on one edge with a lead-pencil or glass rod. The passivity is destroyed by the blow, and the iron is immediately covered with a red film of copper precipitated from the saturated solution of copper nitrate adhering to the iron. If a large sheet of iron is used and the blow is carefully struck, the deposition of copper will proceed from the point of contact and rapidly extend all over the surface of the iron (Fig. 161).

The experiment may be repeated, removing the film of copper by immersing the iron in dilute nitric acid. The

iron may be again rendered passive by immersion in fuming nitric acid.

The sheet iron may be readily prepared by removing the coating of tin on a piece of common tinned iron. The tin plate should be immersed in hydrochloric acid until all the tin has been dissolved.

Sheet iron cleaned and freed from grease; Pt wire; $Cu(NO_3)_2$; fuming HNO_3.

3. Carbon in iron. — The carbon in iron may be separated by dissolving a piece of cast-iron in hydrochloric acid. The insoluble carbon will separate as a black powder. The presence of combined carbon as iron carbide gives rise to the formation of hydrocarbons, which are noticeable by the odor they impart to hydrogen obtained by the action of hydrochloric acid on iron.

Cast-iron; HCl.

4. Combustion of gunpowder and powdered iron. — The comparative combustibility of gunpowder and iron powder may be shown by allowing a mixture of equal weights (one-tenth of a gram of each) to fall from an iron spoon through the flame of burning alcohol. Fifteen cubic centimeters of alcohol are poured into a porcelain evaporating-dish and ignited. As the mixture of gunpowder and iron powder falls through the flame, the iron powder burns with brilliant scintillations, but the gunpowder is not ignited. As the alcohol is burnt out of the dish, the gunpowder remaining in the bottom is finally ignited.

Evaporating-dish; gunpowder; Fe powder; alcohol.

5. Combustion of iron powder and potassium chlorate. — A mixture of 2 parts of iron powder and 1 part of powdered potassium chlorate burns when ignited. The mixture should be placed on an asbestos paper in the hood.

Asbestos paper; Fe powder; $KClO_3$; touch-paper.

6. Reduction of ferric oxide by aluminium. — When aluminium powder is mixed with anhydrous, finely powdered oxides of the metals and the mixture ignited, the aluminium extracts the oxygen from the oxide, forming aluminium oxide and setting free the metal.[1]

This reaction is especially satisfactory when using a mixture of aluminium powder and ferric oxide. The two powders are mixed in equal volumes and ignited with a 2 cm. strip of magnesium ribbon. The mixture burns brightly, leaving a white residue of aluminium oxide. The iron in the finely divided condition immediately burns and reoxidizes.

If a quantity of the mixture of the two powders is placed in a crucible and ignited from the top, the reoxidation of the iron is not so immediate.

$$Fe_2O_3 + 2\ Al = Al_2O_3 + 2\ Fe.$$

Fe_2O_3 powder; Al powder; Mg ribbon.

7. Action of light on potassium ferricyanide. — The reduction of potassium ferricyanide by light is used in photography to produce "blue-prints."

A solution of the salt is mixed with a solution of "citrate of iron and ammonia" and unsized paper is coated with the liquid. When dry the paper is sensitive to light, and if a piece is exposed to bright sunlight in a photographic printing frame and subsequently washed in very dilute hydrochloric acid, a coating of Prussian blue will be left on it. By using a design cut out of paper or a photographic negative, the design or picture may be reproduced.

Photographic printing frame; unsized paper; design or photograph negative; citrate of iron and ammonia; $K_3Fe(CN)_6$.

[1] This reaction is the basis of a series of special experiments recently devised by Goldschmidt. The apparatus and materials are to be had of any dealer in chemical supplies.

COBALT AND NICKEL

1. Deposition of cobalt by magnesium. — A piece of bright, clean magnesium ribbon, when immersed in a solution of cobalt sulphate, becomes covered with a blue-black deposit of metallic cobalt. The salt solution should not contain too much free acid.

$$CoSO_4 + Mg = MgSO_4 + Co.$$

$CoSO_4$ solution ; Mg ribbon.

2. Dehydration of cobalt chloride on filter-paper. — Filter-paper saturated with cobalt chloride and exposed to the action of dry air undergoes a change in color, so much so that the tint is an approximate indication of the moisture content of the air.

Two pieces of filter-paper, which have been previously soaked in concentrated cobalt chloride solution and allowed to dry in the air, are suspended by means of a piece of thread and a bit of wax to the interior of each of 2 bell-jars (Fig. 162). The jars are placed on plates, the one over a crystallizing dish containing water, the other over a crystallizing dish containing anhydrous calcium chloride. The calcium chloride abstracts the moisture from the air, and the paper soon acquires a

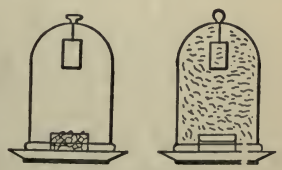

Fig. 162

deep blue color, while the paper over the vessel of water is unaffected and remains red. On interchanging the 2 bell-jars the colors are changed on both papers.

2 bell-jars; 2 plates; 2 crystallizing dishes; $CaCl_2$ (anhydrous); $CoCl_2$.

3. Sympathetic ink. — A word is written on a piece of white paper with dilute cobalt chloride solution. When dry the word is not visible. On heating high above a Bunsen flame the word appears in blue lines, which gradually fade on cooling.

4. Preparation of potassium cobalt nitrite (Fischer's salt). — A characteristic reaction of cobalt, whereby it may be readily distinguished from nickel, is its formation of a yellow crystalline precipitate (potassium cobaltic nitrite) with a solution of potassium nitrite.

Cobalt nitrate solution is acidified with acetic acid and then a solution of potassium nitrite added. On standing over night a yellow crystalline precipitate of the double nitrite will be formed.

$Co(NO_3)_2$; KNO_2.

5. Reduction of nickel oxide by hydrogen. — The reduction of nickel oxide and the pyrophoric nature of the finely reduced metal is well shown by heating the oxide in a short length of combustion-tubing in a current of pure hydrogen. When the reduction is complete, a one-holed cork bearing a short piece of glass tubing should be inserted in the open end of the combustion-tube and the nickel allowed to cool in a current of hydrogen.

The color change from green nickel oxide to black nickel is so marked that the green rather than the black oxide should invariably be used.

A small portion of the still hot metal remaining in the tube after the reduction is complete will burn when allowed to fall through the air.

Finely divided nickel, resulting from the reduction of the oxide, is readily oxidized in the air, and while, when cold, it does not burn with the brilliancy of pyrophoric lead (Ex. 1, p. 400), sufficient heat will be generated on exposing the metal to the air to ignite a small heap of gunpowder.

A few grams of gunpowder are placed on a square of asbestos paper, and the cold reduced nickel poured out of the tube upon it.

If a small quantity of the reduced metal is poured upon the heads of 2 or 3 matches, they will also become ignited.

$$NiO + H_2 = H_2O + Ni.$$

H generator; combustion-tube; 4-tube burner; NiO; gunpowder.

6. Deposition of nickel by electrolysis (nickel plating). — A polished strip of sheet copper is connected to the zinc pole of a bichromate battery and immersed in a solution of ammonium nickel sulphate. The negative pole may be a piece of nickel or platinum. On closing the electric circuit, a gray deposit of nickel will be formed on the copper electrode.

Bichromate battery; sheet Cu; Pt; Ni; ammonium nickel sulphate.

7. Oxidation of nickel in the air. — A sheet of polished nickel, when heated in the air, becomes covered with a thin iridescent coating of the oxide.

8. Solubility of nickel sulphide in sodium sulphide. — Hydrogen sulphide added to an alkaline solution containing nickel produces a deep black solution without the formation of a precipitate.

Nickel chloride solution is treated with a small quantity of tartaric acid, and then sufficient sodium hydroxide solution is added to make the liquid feel soapy. On conducting a current of hydrogen sulphide through the solution, a jet-black solution is formed, which, even on boiling, does not yield a precipitate. On pouring the liquid upon a filter, the black solution runs through the paper, and on washing the filter with hot water the absence of any precipitate may be seen.

This reaction furnishes an excellent means of distinguishing nickel from cobalt in solutions, for with cobalt the sulphide is precipitated, the supernatant liquid remaining colorless.

H_2S generator; solutions of tartaric acid and $NiCl_2$.

9. Preparation of nickel tetracarbonyl by the action of carbon monoxide on reduced nickel. — Carbon monoxide, when conducted over finely reduced nickel, unites with the metal at the ordinary temperatures, forming nickel tetracarbonyl. The vapor passes along with the excess of carbon monoxide, and may be condensed to a colorless liquid if conducted through a tube immersed in a freezing-mixture.

The nickel for this experiment should be very finely divided, and is best obtained by reducing nickel oxide mixed with fibrous asbestos and loosely packed in a 40 cm. length of combustion-tubing. The finely powdered nickel oxide should be thoroughly mixed with the asbestos, to which a large quantity of the powder will cling. Hydrogen, purified by being passed through a solution of potassium permanganate, is conducted through the tube, which is then heated to low redness. As the reduction is effected, the green nickel oxide becomes converted to black nickel. When no more water vapor escapes from the tube, the re-

duction may be considered complete, and the nickel is allowed to cool in a current of hydrogen.

Carbon monoxide from the generator (Ex. 12, p. 298) is conducted through a calcium chloride drying-tube and then directly through the tube containing the reduced nickel. The issuing gas is passed through a long glass elbow extending nearly to the bottom of a test-tube fitted with a two-holed cork. The gas issuing through a shorter elbow in the second hole of the cork should be conducted into the

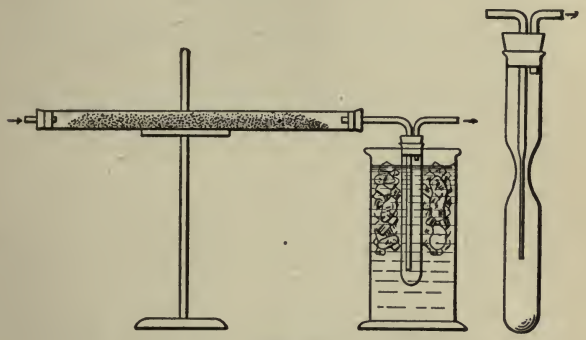

FIG. 163

flue. The test-tube should be immersed in a freezing-mixture of salt and ice (Fig. 163).

As the carbon monoxide comes in contact with the nickel, the temperature of the tube is materially increased, indicating the chemical action. The gas escaping from the glass elbow in the test-tube consists of carbon monoxide containing a quantity of uncondensed vapor of nickel tetracarbonyl, which, owing to its poisonous character, should not be inhaled. The preparation of any considerable quantity of the tetracarbonyl requires too long a time for demonstration on the lecture table, though, if the operation is begun before the hour, 2 or 3 cc. may be obtained. If the test-tube

is somewhat constricted in the middle, the condensed liquid may be sealed off in the glass tube.

If the escaping gas is conducted into the base of a Bunsen burner, the flame becomes brilliantly luminous from the incandescence of fine particles of nickel. The structure of the Bunsen flame is markedly shown by the varying intensity of brilliancy in its different parts.

The escaping gas may be ignited at the end of a glass jet, where it will burn brightly, giving a green smoke consisting of nickel oxide. If a white porcelain dish is held in the upper portion of the flame, a green deposit of nickel oxide will be obtained, while, if the dish is depressed on the flame, black metallic nickel will be deposited.

The nickel tetracarbonyl vapor is easily decomposed by heat into nickel and carbon monoxide, and consequently, if the glass tube through which the gas is being conducted is strongly heated, a black metallic deposit of nickel will be obtained, and the luminosity of the flame will be diminished until ultimately the blue flame of carbon monoxide alone is obtained. By gently heating the tube, a mirror possessing a brilliant metallic lustre will be formed. The brilliancy of the mirror is a function of the temperature, and if the tube is heated to about 200° C., the best results are secured. By attaching a Y-tube to the tube conducting the waste gases the gas may be suddenly switched into a glass tube which has been heated to 185° in an air-bath. Instantly the interior of the tube becomes coated with a nickel mirror.

$$Ni + 4\ CO = Ni(CO)_4.$$

4-tube burner; 40 cm. length combustion-tubing; test-tube, cork, and elbows; porcelain dish; air-bath and thermometer; fibrous asbestos; CO generator (Fig. 120, p. 299); H generator; gas washing-bottle containing $KMnO_4$ solution; NiO; ice and salt freezing-mixture.

APPENDIX

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Description of the hydrogen sulphide generator in use in the laboratory of Wesleyan University: —

"It consists principally of three glass bottles, *A*, *B*, and *C*, each provided with an orifice near the bottom. *A* is the generator proper, whose mouth is wide so as to admit large lumps of iron sulphide, and whose height is about three times its diameter. It has a capacity of about 16 liters.

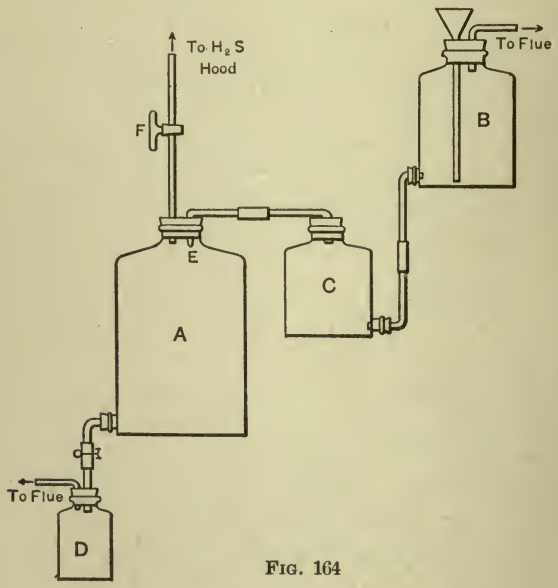

FIG. 164

Through the rubber stopper which closes its mouth passes the gas main, controlled by the main cock, *F*, and terminating at a suitable hood in as many distributing cocks as may be desired. Students have access to the distributing cocks only. Through the same stopper enters the acid-supply tube, drawn down at the end *E* to a jet capable of delivering

a stream of acid not thicker than the shank of an ordinary pin. This jet projects as little as possible below the stopper. Through the tubulure at the bottom, with its inner end just flush with the stopper, passes a rather thick-walled tube controlled below by a pinch-cock, as shown. By means of this tube the spent acid is delivered either into a sink or waste-pipe capable of thorough flushing, or into a small bottle, *D*, connected with a flue, which may be detached just below the pinch-cock and emptied as required. The generator bottle is charged with large pieces of iron sulphide until completely full, and *should not be allowed to become less than half full* before recharging. The spent acid must never be allowed to accumulate in such quantities as to reach the top of the sulphide.

"*B* is the acid reservoir, wide in proportion to its height, with a capacity of about 8 liters. The stopper at its mouth admits a funnel whose neck is continued by a glass tube passing to the bottom of the bottle. This reservoir is connected with a flue as shown. The acid used is commercial hydrochloric, diluted with an equal volume of water. A proper charge of acid is the amount necessary to fill *B* half full when acid is running from, or stands at, the jet *E*. The height at which the reservoir itself is fixed depends, of course, largely on the pressure to be overcome by the gas. A difference of level of 6 inches between the bottom of *B* and the top of *A* has been found ample for ordinary analytical work.

"*C* is the gas reservoir, designed chiefly to retain whatever gas is made after the distributing cocks, or *F*, are closed. Like *B*, it is wide and low. It has a capacity of about 4 liters. Its mouth should be on exactly the same level as that of *A*, and the vertical portions of the tube connecting *C* and *A* are made as short as possible.

"The tubing used in the apparatus is of glass. The rubber

stoppers are protected with a thin coating of paraffin, crowded into place while the latter is still warm and soft, and firmly wired. The rubber connections are coated within with paraffin (by pouring hot paraffin through them when perfectly dry), and securely wired."

(W. P. Bradley, in *Am. Chem. Journal*, XXI., 370.)

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